1
Amino Acids

BY WENG C. CHAN and AVRIL HIGTON

1 Introduction

This chapter covers the newly published chemistry of amino acids for the year 2000; some biological aspects are also included to accompany the relevant chemical studies. Some references from 1999 stray into the list as do a few from early 2001. Literature citations were selected through Chemical Abstracts (Volume 132, Issue 6 to Volume 133, Issue 8) and through major journals, which frequently contain amino acid-related papers. Papers on related material have again been grouped without comment. Conference proceedings are largely excluded, and no patent material has been included.

Amino acid analogues containing oxy acids of phosphorus, and boron and sulfonic acids have been included in the section relevant to the type of amino acid considered, as have the increasing numbers of metal complexes containing amino acid ligands. References have been included in the section relevant to the primary theme of the paper, even if other aspects are also included.

2 Textbooks and Reviews

The reviews listed here only relate to general amino acid topics, specific reviews are mentioned in the appropriate section. Textbooks have appeared describing a practical approach to amino acid derivatives,[1] to amino acids as natural products,[2] and to phosphorus analogues of amino acids.[3]

Total syntheses of amino acids to the year 1998 have been reviewed.[4] Reviews of the syntheses of amino acids using biochemical, biosynthetic or a combination of chemical and biotechnological methods have been reported; the synthesis of amino acids by a combination of chemical and biochemical processes[5] and biotechnology[6] and biocatalytic production of atypical amino acids.[7] Engineering microbial pathways for amino acid production have been reviewed.[8] The stereoselective syntheses of amino acids using hydantoinases and carbamoylases have been reviewed,[9] as have new routes to chiral amino acids using biosynthetic pathways.[10] Reviews of asymmetric syntheses that do not fit comfortably into other sections have included; asymmetric synthesis of unnatural amino acids using commercially available chiral nonracemic glycinates[11] and the asymmetric

Amino Acids, Peptides and Proteins, Volume 33

synthesis of α-amino acids and β-amino acids using chiral zirconium complexes as catalysts.[12] The thermodynamics of the asymmetric synthesis of amino acids have also been reviewed.[13] Reviews have also appeared on the progress of the commercial synthesis of amino acids and other nutraceuticals,[14] on approaches to the total synthesis of natural product-based compound libraries using polymeric supports,[15] on tracer amino acids for the investigation of protein and amino acid metabolism in humans,[16] of the synthesis of the unusual amino acids found in peptides of aquatic origin and their incorporation into the peptides,[17] and on the uses of β-amino acids in medicinal chemistry and as building blocks for peptide modification have been reviewed.[18]

The etymology of amino acid names has been reviewed.[19]

3 Naturally Occurring Amino Acids

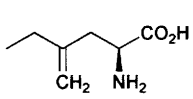
Studies have been reported on the composition and abundance of amino acids in ores from the supergene zones of Mingshan gold ore deposit,[20] and in the hydrothermally altered sediments from the Juan de Fuca ridge in the pacific ocean. Both the free and hydrolysable amino acid composition was analysed.[21] Naturally occurring aminophosphonic acids have been reviewed.[22]

3.1 Occurrences of Known Amino Acids. – Glutamic acid, glutamine, pyroglutamic acid and arginine have been isolated from the pronotal and elytral secretions of *Platyphora opima* and *Desmogramma subtropica* along with triterpene saponins and phosphatidylcholines.[23]

The amino acid *N'*-[(*R*)-1-carboxyethyl]-N^{α}-(D-galacturonyl)-L-lysine has been identified as a component of the O-specific polysaccharide of *Proteus mirabilis*.[24]

3.2 New Naturally Occurring Amino Acids. – (*S*)-2-Methylglutamine and (*S*)-5-methylarginine have been identified in the active site region of methyl-coenzyme M reductase. The biosynthesis of these and other methylated amino acids is discussed, together with the implication for the production of methane greenhouse gas.[25]

Sponges continue to be fertile ground for the discovery of novel amino acids; three new *N*-acyl-2-methylene-β-alanine methyl esters, hurghamids E–G, have been isolated from *Hippospongia* spp.[26] The amino acid (**1**) has been isolated from the Caribbean sponge *Plaktoris* simplex along with plakortones and simplactones.[27] The bromotyrosine compound (**2**) was isolated as a secondary metabolite from the sponge *Verulonga gigantea* and identified from spectral data,[28] and herbacic acid (**3**) has been isolated from the sponge *Dysidea herbacea*.[29] Synthetic studies have also been carried out.[30] The structure of pulcherrimine, a bitter-tasting amino acid from the sea urchin *Hemicentrotus pulcherrimus* was elucidated as (**4**) from chemical and spectral data.[31] Makalika ester and makalikone ester (**5**, X = CH_2 or CO, respectively) were isolated from the sea hare and their structures determined from spectral data.[32]



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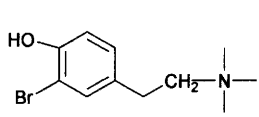

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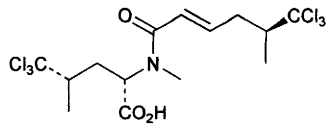

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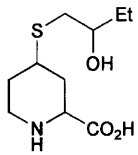

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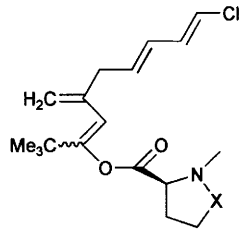

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3.3 New Amino Acids from Hydrolysates. – The structures of microscleridermins F–I, isolated from the sponge *Microscleroderma* sp., were elucidated from chemical and spectral data. The compounds incorporate an unusual long chain β-amino acid.[33]

4 Chemical Synthesis and Resolution of Amino Acids

Readers seeking syntheses of particular amino acids should consult both Sections 4 and 6.3 of this chapter.

Two reviews on the synthesis of conformationally constrained aromatic amino acids are available.[34,35] Reviews of the preparation of aziridine carboxylates, carboxamides and lactones and their transformation into α- and β-amino acids,[36] and the synthesis of vinyl amino acids[37] have been published. A novel synthetic protocol for enantiomerically pure substituted prolines[38] has been reported. The synthesis of unnatural amino acids by reduction and ozonolysis of aromatic amino acids has been reviewed,[39] and on the synthesis of non-natural α-amino acid derivatives.[40] Recent advances in the synthesis and application of labelled nucleic acids, amino acids and carbohydrates,[41] and a review with eighteen references, including the preparation of Schiff bases, tandem reduction of and alkylation of Schiff base esters and the synthesis of complex amino acid polyols,[42] have been published.

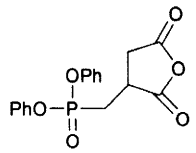
4.1 General Methods for the Synthesis of α-Amino Acids, Including Enantioselective Synthesis. – Recent synthetic advances in the preparation of phosphorus analogues of α-amino acids[43] and a rapid solid phase synthesis of non-proteinogenic *N*-acetyl α-amino acids[44] have been reported. A review (with forty-one references) examining practical catalysts and processes for the synthesis of both L- and D-amino acids using ligand systems derived from D-glucose has been published.[45]

The enthalpy of formation of peptides compared with values for the parent

amino acids has been reviewed. The review also covers values for amides compared to carboxylic acids.[46]

Synthetic routes for the preparation of the model compound *e.g.* (**6**), an analogue of phosphoryl amino acids,[47] α-amino alkanephosphonic acids[48] and ω-aminophosphonic acids[49] have been described. The syntheses of compounds with side chain C–P links[50] and asymmetric synthesis[51] have been reviewed. A review of pentacoordinated phosphorus compounds of amino acids and nucleosides has been published.[52]

A procedure for the synthesis of (*R*)- and (*S*)-enantiomers of α-carbon deuterium-labelled α-amino acids has been described. The labelled enantiomers were resolved on a chiral ion exchanger.[53] The configuration of Aeruginosin 298-A (**7**) has been reassigned based on its total synthesis, incorporation of D-leucine gave the natural product.[54]



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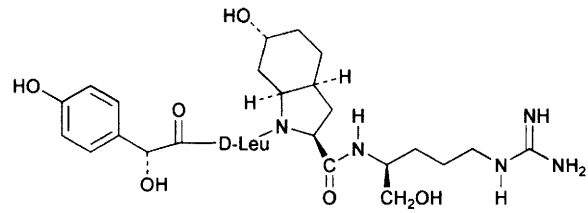

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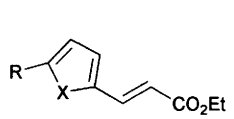
4.1.1 Amination of Alkanoic Acid Derivatives by Amines and Amine-related Reagents. Amino acid syntheses using an amination route, including a one-pot synthesis by reaction of 2-hydroxy-2-methoxyacetic acid methyl ester with benzyloxyamine and an alkyl radical using stannyl radical-mediated reaction,[55] the asymmetric synthesis of fluoro amines and amino acids using reducing agent-free, reductive amination of fluorocarbonyl compounds in three simple steps,[56] and the synthesis of chiral C-protected α-amino aldehydes of high optical purity[57] have been reported. The synthesis of enantiometrically pure α-amino acid derivatives from aldimines and tributyltin cyanide or achiral aldehydes, amines and hydrogen cyanide using a chiral zirconium catalyst,[58] and a convenient synthesis of the new sugar amino acid, 3-aminomethyl-3-deoxy-1,2-*O*-isopropylidene-α-D-ribo-hexofuranuronic acid,[59] have been described. It has been demonstrated that furyl and thienyl acrylates (**8**) (X = O, S; R = H, Me) could be subjected to aminohydroxylation with high selectivity, but pyrrolyl acrylates resist aminohydroxylation under the present reaction conditions. The resulting amino hydroxylation products (**9**) were readily converted to β-hydroxy-α-amino acids.[60]

Guanidinium- and amine-containing amino acids based on a proline or alanine scaffold have been prepared. The guanidinium compounds were best prepared using 1*H*-pyrazole-1-carboxamidine hydrochloride as the guanidinating reagent, and the installed guanidino-group protected with Pmc. The resulting amino acids were incorporated into oligopeptides and tested for Tat-TAR interaction.[61] A method for the synthesis of ethylene-bridged (N^{δ} to N^{ω}) analogues of arginine (**10**) has been given. The initial step of the synthesis involved



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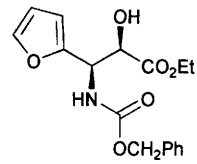

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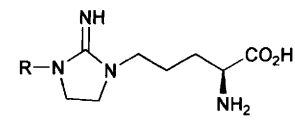

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the preparation of (*S*)-2-azido-5-bromopentanoic acid.[62] Large-scale syntheses of unnatural amino acids have been achieved by amination of keto acids using transaminases in a whole cell biotransformation,[63] and a convenient, scaleable process for the preparation of substituted phenylglycines by a modified Strecker reaction. Bisulfite-mediated addition of benzylamine and cyanide anion to substituted benzaldehydes gave aminonitriles, which were hydrolysed to the N-protected amino acid. Debenzylation resulted in good yields of substituted phenylglycines.[64]

4.1.2 Carboxylation of Alkylamines and Imines and Related Methods. A catalytic enantioselective aza-Diels–Alder reactions of imines to give optically active non-proteinogenic α-amino acids has been described.[65]

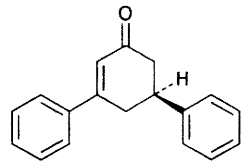
4.1.3 Use of Chiral Synthons in Amino Acid Synthesis. A stereocontrolled synthesis has been reported of enantiopure hydroxylamines having additional functionalities by reaction of chiral nitrones with a variety of nucleophiles. The hydroxylamines can be converted into amino acids and other nitrogenated compounds.[66] Cinchonidine and cinchonine, *N*-alkylated with Merrifield resin, have been employed as phase transfer catalysts for the enantioselective alkylation of enolates from *N*-(diphenylmethylene) glycine esters. Cinchonidine gave rise to (*S*)-isomers, whereas cinchonine gave (*R*)-isomers of amino acids.[67] The importance of a spacer in new chiral phase transfer supported catalysts used for the asymmetric synthesis of α-amino acids was studied. Polymer-supported cinchona alkaloid salts with different spacers were used as phase transfer catalysts in the asymmetric *C*-alkylation of *N*-diphenyl methylene glycine t-butyl ester for the synthesis of phenylalanine. Best results were obtained with cinchoninium iodide bound to polystyrene with a four-carbon spacer.[68] Syntheses of acyclic and heterocyclic (*S*)- and (*R*)-α-amino acids have been prepared from 1,5-dimethyl-4-phenylimidazolidin-2-one derived iminic glycinimides.[69]

Threo-β-hydroxy-1-glutamic acid derivatives with different carboxyl protecting groups have been synthesised using an aziridine-2-carboxylate as a chiral synthon in an aldol reaction[70] and protected (*S*)-4-carboxytetrahydro-1,3-oxazin-6-ones, synthesised by Baeyer–Villiger reaction on 4-ketoproline, have been developed as chiral templates in the synthesis of β-substituted aspartic acids.[71] α,α-Disubstituted α-amino acids have been synthesised asymmetrically under mild conditions using oxazinone and pyrazinone derivatives as chiral reagents,[72] while α,α-dialkyl-α-amino acids have been synthesised by enantioselective solid–liquid phase transfer catalytic alkylation of the aldimine Schiff base of amino acid *tert*-butyl esters with chiral quaternary ammonium bromides.[73]

Adducts from the diastereoselective Mannich-type reactions of aldehydes, 2-furylboronic acid and the chiral amine template (*S*)-5-phenylmorpholin-2-one have been used in the synthesis of enantiomerically pure D-α-amino acids.[74] L-2′-Bromo-phenylalanine and -tyrosine have been prepared using (−)-2,10-camphor sultam as a chiral auxiliary.[75] A copper(II) (salen) complex was used as an asymmetric phase transfer catalyst for the *C*-alkylation of *N*-benzylidene alanine methyl ester in the synthesis of α-methyl-α-amino acids; the enantiomeric excess was up to 86%.[76] Asymmetric syntheses of (2*R*,3*R*)- and (2*R*,3*S*)-3-hydroxypipecolic acids have been reported; the key step was the addition of Buchi's Grignard reagent to a chiral serinal.[77]

γ-Fluorinated-α-amino acids have been synthesised using 2-hydroxy-3-pinanone as a chiral auxilliary.[78] The asymmetric synthesis of quaternary α-amino acids using D-ribonolactone acetonide as a chiral auxiliary is presented.[79]

The preparation of planar chiral mimetics and their use in the stereoselective catalysis of the addition of Et_2Zn to PhCHO resulting in (*R*)-PhCH(Et)OH is described.[80] A new route for the preparation of enantiomerically pure quaternary α-substituted serine esters, involving the diastereoselective functionalisation of an oxazolidine ester enolate having an exocyclic chiral appendage is given.[81] The asymmetric synthesis of an (*S*)-ornithine and a chiral 2-cyclohexenone (**11**) *via* an enantioselective Michael reaction using chiral ammonium salts is reported.[82]



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An easy three step process for the synthesis of optically pure α-amino acid derivatives bearing a bulky α-substituent involving an external chiral ligand-mediated asymmetrical addition of phenyllithium to an anisidine amine, oxidative removal of a N-PMP group, and finally oxidative conversion of the Ph group to a carboxyl group is reported.[83]

4.1.4 Use of Rearrangements Generating a Carbon–Nitrogen Bond. Pentacoordinated phosphorus compounds of amino acids and nucleosides,[84] the synthesis of compounds with side chain C–P links[85] and the asymmetric synthesis of phosphorus analogues of amino acids[86] have been reviewed.

Trisubstituted-benzoyl aziridine carboxylates have been synthesised from α-alkyl-β-amino acids. Ring expansion or ring opening of these compounds lead to α-substituted α-hydroxy-β- and β-hydroxy-α-amino acids.[87] Syn and anti-β-substituted α-amino acids have been prepared by a nitrone cycloaddition approach.[88] Various fluorenyl imines undergo a catalytic asymmetric Strecker-type reaction with trimethyl silyl cyanide in the presence of a Lewis acid–Lewis base bifunctional catalyst and a catalytic amount of phenol. The products were converted to amino acids without loss of enantiomeric purity.[89] Oxidative

rearrangement of azabicyclo[2.2.1]heptenes with mCPBA generated the oxazabicyclo[3.2.1]octenes, precursors for hydroxylated cyclopentyglycines.[90]

O-Phosphoryl amino acid esters have been prepared from *N*,*N*-dialkylphosphoramidates and the side chain hydroxy groups of tyrosine, serine and threonine.[91] *N*-(*O*,*S*-dimethylthiophosphoryl)-α-amino acid esters have been prepared in high yield and optical purity, and tested for insecticidal activity.[92]

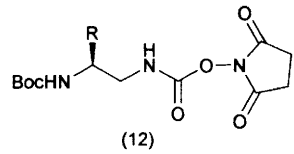
4.1.5 Other Rearrangements. Other rearrangement reactions have also been reported. The allylation of α-amino acid esters has been shown to give rise to intermediate quaternary ammonium salts which undergo proton abstraction to give ylides and [2,3]-Stevens sigmatropic rearrangement to give α-allyl α-amino acids,[93] allylic esters of TFA-protected amino acids underwent asymmetric Claisen rearrangements in the presence of cinchona alkaloids giving rise to γ,δ unsaturated amino acids in a highly stereoselective fashion.[94] The stereoselective synthesis of allylic amines by rearrangement, by the heating in xylene, under reflux, of allylic trifluoroacetimides allowed the synthesis of polyoxamic acid and derivatives of other α-amino acids.[95]

A process for the chelate–enolate Claisen rearrangements has also been reported for the asymmetric synthesis of unsaturated amino acids and peptides[96] and Claisen rearrangement methodology has also been reported for the synthesis of (2*S*,3*S*)-, (2*S*,3*R*)-substituted-L-glutamic acids starting from D-serine.[97] Unnatural α-amino acids were prepared when azetidin-2,3-diones have been reacted with primary amines in a one-step synthesis[98] and a novel synthetic route for the preparation of labelled amino acids by the rearrangement of α-aminocyclopropanone hydrate[99] and the synthesis of α-amino esters *via* the radical reaction of phenylsulfonyl oxime ethers on a solid support[100] have been described.

The synthesis of α- and β-amino acids by the isomerisation of aziridinyl ethers using superbases has been described.[101] The first racemic synthesis of the non-proteinogenic amino acid, (2*S*,3*R*,4*R*)-4-hydroxy-3-methyl-proline has been achieved *via* indolactonisation of an unnatural amino acid derivative. The relative stereochemistry was derived from an efficient silicon assisted aza-[2,3]-Wittig sigmatropic rearrangement.[102] Ring-opening of N-(PhF)serine-derived cyclic sulfamidate has been achieved with different nucleophiles (β-keto esters, β-keto ketones, dimethyl malonate, nitroethane, sodium azide, imidazole and potassium thiocyanate) to prepare a variety of amino acid analogues – two different pathways for ring opening were elucidated by the authors.[103] The application of Curtius rearrangements for the simple conversion of a number of N-Boc-protected β-amino acids into the corresponding *O*-succinimidyl-2-(tert-butoxycarbonylamino)ethylcarbamate derivatives (**12**, R = H, Me, I-Pr, CH_2Ph, CH_2CO_2Ph) has been reported.[104]

4.1.6 Amidocarbonylation and Related Multicomponent Processes. An overview of transition metal-catalysed amidocarbonylation together with views on future synthetic developments has been recently published.[105]

Biologically significant molecules have been prepared using this route of synthesis. N-Boc-iturinic acid and 2-methyl-3-aminopropanoic acid, compo-



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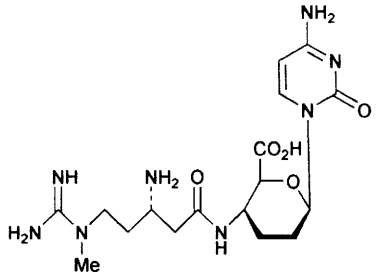
nents of the antifungal peptide iturin and depsipeptide cryptophycin, have been prepared by alkylation of functionalised succinic acid.[106] Blastidic acid, a component amino acid of the antibiotic blasticidin S (**13**), has been synthesised for the first time from α,γ-diaminobutyric acid by carbon-chain elongation, *N*-methylation and followed by amidination using *O*-methyl-*N*-nitroisourea.[107]

Galactopyranosyl azide was esterified with allyl tetramethylazelaoyl chloride and attached to Wang or Merrifield polymer through the other acid group. The azide was then reduced to amine using $HS(CH_2)_3SH$ and subjected to four-component reaction with aldehyde, isocyanate and HCO_2H. Acid hydrolysis yielded substituted phenylglycinamides.[108]



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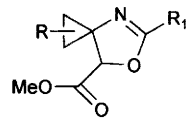

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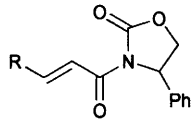
β^{2,2}-HBip, a biphenyl-substituted 3-amino-2,2-dimethylpropanoic acid has been prepared and converted into β-homo-peptides,[109] and 2-chloro-2-cyclopropylideneacetates, treated with carboxamides under basic conditions, was shown to undergo a domino transformation involving a Michael addition followed by an intramolecular nucleophilic substitution to afford 4-spirocyclopropane-annelated oxazoline-5-carboxylates [**14**, R = H, Et, $(CH_2)_2OCH_2Ph$, R_1 = Ph, nicotinic acid amide, furan-2-carboxamide, Me, Et, Pr, $C(CH_3)_3$]. These compounds are protected α-hydroxy-β-amino acids.[110] *Syn*-γ-hydroxy-β-amino acids have been prepared stereoselectively by iodolactonisation of 3-amino-4-pentenoic acid,[111] and successive protection, Arndt–Eisert reaction, Wolff rearrangement and deprotection of L-valine has been shown to lead to L-amino-4-methyl valeric acid hydrochloride.[112] The preparation of achiral and of enantiopure geminally disubstituted β-amino acids for β-peptide synthesis has been described.[113]

Previous reports in the literature that the treatment of *N*,*N*-dibenzyl amino alcohols with sulfonyl chloride lead to tetrahydroisoquinolines have been disproved. The products, which are intermediates in the synthesis of β-amino acids, are in fact β-chloro amines.[114]

Aliphatic α-amino acids have been synthesised by one-pot reaction of aldehydes, KOH, ammonia and $CHCl_3$ in the presence of urea as a reversible phase transfer catalyst.[115]

4.1.7 From Glycine Derivatives and Imines of Glycoxylic Acid Derivatives. The synthesis of amino acids from glycine derivatives has been reported. The oxazolidinones (**15**) (R = various alkyl or aryl) served as Michael acceptors in addition reactions with achiral Ni(II) complexes of glycine Schiff bases. Deprotection of the appropriate resulting nickel complex (II) resulted in *e.g.* pyroglutamic acid in 96% yield with virtually complete stereoselectivity.[116]

Enantiopure 3-substituted pyroglutamic acids have been synthesised by Michael addition between 4*R*- or 4*S*-(*N*-*trans*-enoyl)oxazolidinones and the Ni(II)-complex of the chiral Schiff base of glycine with (*S*)-*o*-[*N*-(*N*-benzylprolyl)amino]benzophenone,[117] a Schiff base protected glycine, supported on poly(ethylene glycol), was reacted with electrophiles under microwave activation to produce α-amino acids,[118] and enantiomerically pure 2′,6′-dimethyltyrosine was synthesised by reaction of 4′-benzyloxy-2′,6′-dimethylbenzyl bromide with Ni(II) complexes of the chiral Schiff base of glycine with (*S*)-α-[*N*-(-benzylprolyl)amino]benzophenone.[119]



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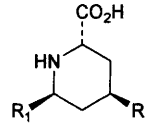

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The diasteromerically pure pipecolic acids (**16**, R = H, Me, OH; R_1 = Pr, Et) have been synthesised from (*S*)-2-phenylglycinol[120] and imidazolidinone-bound glycine enolate derivatives were shown to undergo aldol condensation with aldehydes β-hydroxy-α-amino acids in a two-step process.[121]

N-Acyl-α-triphenylphosphonioglycinates, when reacted with carbon nucleophiles, gave rise to α-functionalised glycine derivatives.[122] New (*Z*)-α,β-didehydroamino acid derivatives with 3,5-dihydro-2*H*-1,4-oxazin-2-one structure have been synthesised by condensation of the chiral glycine equivalent with aldehydes in the presence of K_2CO_3 under mild solid–liquid phase transfer catalysis reaction conditions.[123]

Imines of glycoxylic acid derivatives have also been employed in the synthesis of amino acids. Alkylations and condensation reactions of both glycine and alanine imine have been studied and were shown to give rise to enolates which could be alkylated in a highly diastereoselective manner. The reactions gave rise to mono and dialkylated α-amino acids and heterocyclic derivatives.[124]

Amino acids derivatives have been synthesised *via* a scandium triflate three-component reaction of phenols, glyoxylates and amines,[125] pre-protected α,α-disubstituted amino acids have been prepared asymmetrically from tert-butylsulfinyl ketimines,[126] α- and β-amino acids by the stereoselective alkylation of chiral glycine and β-alanine derivatives[127] and after hydrolysis, α-methyl α-amino acids by the diastereoselective alkylation of an iminic alanine template with a 1,2,3,6-tetrahydro-2-pyrazinone structure.[128] The first asymmetric synthesis of α-amino acids based on diastereoselective carbon radical addition to glyoxylic imine derivatives is reported.[129]

4.1.8 From Dehydro-amino Acids Derivatives. The synthesis of β-substituted-α,β-dehydroamino acids by a Michael addition of heterocyclic nucleophiles to the methyl esters of *N-tert*-butoxycarbonyl-*N*-(4-toluenesulfonyl)-α,β-dehydro-amino acids followed by a base-induced elimination of the 4-toluenesulfonyl group with the regeneration of the α,β-double bond has been reported,[130] and that of cyclic amino acid derivatives by a ring closing metathesis reaction on soluble poly(ethylene glycol)-supported allylglycine derivatives.[131]

Copper-promoted reaction of serine-derived organozinc reagents with allylic electrophiles gave Fmoc-protected amino acids ready for peptide synthesis[132] and new rhodium catalysts with unsymmetrical P-chirogenic bis(phosphino)ethanes, BisP*-Rh, were shown to exhibit very high enantioselectivity in the hydrogenation of α-dehydroamino acid derivatives.[133]

4.2 The Synthesis of Protein Amino Acids and Other Well-known Naturally Occurring Amino Acids. – Because of the commercial and biological importance of the protein amino acids and other naturally occurring amino acids, much work has been targeted towards the syntheses of these molecules. Work in the field has been the subject of a number of reviews, including the synthesis (and applications) of phenylalanine,[134] the synthesis of L-carnitine,[135] the syntheses (and biological evaluation of) (+)-lactacystin and its analogues,[136] and the asymmetric syntheses of α-substituted serines.[137]

Reviews have also been produced regarding the industrial production of some of these materials. The industrial production of D-alanine and D-tartaric acid using microorganisms has been reviewed,[138] as has the current status of lysine[139] and L-cysteine[140] production in China.

Large-scale processes have been developed for D-pyroglutamic acid production from L-glutamic acid by successive racemisation, resolution and dehydration.[141] and for the synthesis of L-DOPA tert-butyl ester, using catalytic enantioselective phase-transfer alkylation.[142]

The preparation (and applications) of phenylalanine[143] and L-threonine[144] have been reported. The synthesis has also been reported of tritium-labelled thyroxine and related compounds.[145] A study which may contribute to the explanation of the origin of life on earth has been published.[146] In this study, the thermochemical aspects of the conversion of the gaseous system CO_2–N_2–H_2O into a solid amino acid condensate in an electric discharge plasma are considered.

A number of enzymic syntheses have also featured in the literature including the enzymic synthesis of ^{32}P-labelled phosphoarginine,[147] the preparation and resolution of both epimers of L-cyclopentenylglycine (this method was used for preparation of ^{13}C-labelled compounds for use as tracers)[148] and the synthesis of L-tryptophan from L-cysteine and indole using the genetic engineering strain WW-1l.[149] The two isotopomers of L-phenylalanine, ^{13}C or ^{14}C labelled in the carboxyl group have also been synthesised enzymatically,[150] as has L-[4-^{13}C] aspartic acid.[151] [^{15}N]-D-isovaline was prepared from DL-[α-^{15}N]-α-aminoisovaleramide by enzymic resolution with *Mycobacterium neoaurum*.[152]

The synthesis of common naturally occurring amino acids and their derivatives has been widely reported. The effects of the reaction conditions and the

preparation methods of the catalyst and the related technology for the alkali water catalytic oxidation of ethanolamine to glycine using a Cu/ZnO catalyst have been studied.[153] Phenylglycine was synthesised in one pot from benzaldehyde, KOH, NH_4OH and $CHCl_3$ under the catalysis of a phase transfer catalyst and β-cyclodextrin,[154] and (*S*)-cyclohexyl glycine has been prepared in high yield by hydrogenation of (*S*)-phenylglycine using rhodium on carbon as the catalyst.[155] A new synthetic route for the preparation of *p*-hydroxyphenylglycine and some analogues from *p*-benzoquinone has been shown to achieve a diastereoselectivity of 60% using 8-phenylmenthyl acetate as the chiral auxiliary.[156]

The synthesis of enantiomerically pure (*S*)-phenylalanine[157] and the synthesis of phenylalanine from benzylidene glycinate *via* C-alkylation using microwave irradiation and phase-transfer catalysis have been described.[158] An efficient method has been reported for the conversion of β-phenylisoserine to β-hydroxyphenylalanine derivatives *via* aziridines,[159] and highly functionalised phenylalanine derivatives have been prepared using cross-enyne metathesis and Diels–Alder addition as key steps.[160] L-(+)-Homophenylalanine hydrochloride has been synthesised in 55% yield with 99% enantiomeric excess from *N*-phthaloyl-L-aspartic acid.[161]

The syntheses of [ring-^{14}C]-L-tyrosine from [U-^{14}C]-phenol,[162] and other isotopically-labelled amino acids, including 2,3,4,2′,3′,5′,6′-$^{2}H_7$-L-tyrosine[163] and 3,3,4,4,3′,3′,4′,4′-$^{2}H_8$-homocystine,[164] have been reported.

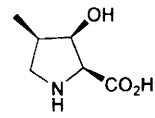
O-Phosphoryl amino acid esters have been prepared from *N*,*N*-dialkylphosphoramidates and the side chain hydroxy groups of tyrosine, serine and threonine.[165] D,L-Serine has been prepared in 89% yield with 92.0% purity in two steps from α-chloro-β-aminoproprionitrile hydrochloride.[166]

A Co(III) imino acid complex has been used for the stereospecific incorporation of deuterium into the α- and β-carbon atoms in α-amino acids[167]and *N*-(α-stannylalkyl)oxazolidinones, prepared in three steps from aldehydes, have been shown to undergo tin-lithium exchange to give *N*-(α-lithioalkyl)oxazolidinones. The latter undergo carboxylation to diastereopure *N*-(α-carboxyalkyl)oxazolidinones. Birch reduction of the oxazolidinone moiety then yielded amino acids; and so this rapid method is useful for the preparation of ^{11}C-amino acids.[168] The synthesis of isotopically labelled L-α-amino acids with an asymmetric centre at C-3 has been reported and the method can be adapted to allow the introduction of a label at each site of L-valine.[169] A synthesis of γ-oxo-α-amino acids from polymer-supported α-imino acetates has been reported.[170]

α-Hydroxy and α-amino acids have been prepared by the nucleophilic ring opening of gem-dicyanoepoxides by LiBr or Li_2NiBr_4 in the presence of hydroxylamines *via* α-halohydroxamic acids.[171]

A novel synthetic protocol for enantiopure substituted prolines[172] and the diastereoselective synthesis of (2*S*,3*S*,4*S*)-3-hydroxy-4-methylproline (**17**), a common constituent of antifungal cyclopeptides, from unsaturated lactams are described.[173]

Stereoselective syntheses of (*S*)-5-hydroxynorvaline from glutamic acid,[174] (−)-*N*-Boc-AHPPA[175] and both enantiomers of *trans*-4-pipecolic acid and the



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natural product (−)-SS20846A have been reported.[176] In the latter study the stereochemistry of key intermediates was established by X-ray diffraction analysis.

A large number of amides and esters of glutamic acid have been prepared using chemoselective ring opening of N-Boc pyroglutamic-Wang resin by heteronucleophiles.[177] and a simple transformation of L- and D-glutamic acids into all four possible stereoisomers of 5-hydroxylysine has also been reported.[178]

(−)-Kainic acid has been synthesised using a titanium-mediated cyclisation sequence starting from L-serine,[179] by a sulfanyl radical addition–cyclisation–elimination reaction of diallylamines in the presence of thiophenol and AIBN[180] and by employing a concurrent Chugaev syn-elimination and intramolecular ene reaction from (+)-*cis*-4-carbobenzoxyamino-2-cyclopentenol.[181] A range of 4-arylsulfanyl-substituted kainoid amino acids have been synthesised from *trans*-4-hydroxy-1-proline.[182]

Synthetic routes have also been reported for a phosphonic analogue of (−)-allo-norcoronamic acid.[183] (+)-alloisoleucine,[184] optically pure L-homocysteine from L-methioine (in an easy two step synthesis)[185] and the naturally occurring (2*S*,3*R*, 4*S*)-3,4-methanoproline and its synthetic constitutional isomers.[186] An efficient method has also been developed *via* the Schollkopf chiral auxiliary for the asymmetric syntheses of iso-, homo- and benzo-tryptophan.[187]

Stereoselective conjugate addition of lithiated (*S*)-(α-methylbenzyl)benzylamide to (*E*)-7-(tosyloxy)hept-2-enoic acid tert-butyl ester, followed by deprotection, gave protected β-homolysine of greater than 99% enantiomeric purity.[188] The syntheses of the following materials have also been reported: (±)-homohistidine was prepared from the readily available urocanate;[189] L-glutamine from L-glutamic acid (*via* a three step synthesis);[190] L-cysteic acid (by electrooxidation);[191] *cis*-3-hydroxy-L-proline from β-alanine;[192] and (3*S*,4*R*)-3,4-dimethylglutamine, by asymmetric Michael addition and electrophilic oxidation – three adjacent stereogenic centres were generated simultaneously in this synthesis using a camphorsultam chiral auxiliary.[193]

Resin bound *N*-acylated amino acid aldehydes were converted in a single step to a-hydroxy phosphonates by a Pudovik reaction and in six steps to hydroxystatine amides, useful for constructing multiple aspartic acid transition state isosteres.[194] The synthesis of a series of L-alanine hydroxamate sulfonylated derivatives as protease inhibitors has been reported.[195] The compounds were tested as inhibitors of *Clostridium histolyticum* collagenase.[196] Protected 4-hydroxypyroglutamic acids were prepared by 1,3-cycloaddition of furfuryl nitrones with acrylates.[197] The stereoselective synthesis of both enantiomers of *threo*- and *erythro*-β-hydroxy norvaline, involving the addition of different organometallics to (*S*)-serine derivatives, has been reported,[198] whereas three approaches to the synthesis of L-leucine selectively labelled with carbon-13 or deuterium in either

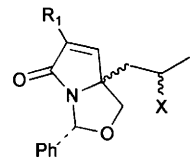
diastereotopic methyl group have been followed. In all three methods the stereogenic centre at C-2 was created with total stereocontrol.[199] A new method has been reported for the synthesis of 2-phenylproline by intramolecular cyclisation of *N*-(3-chloropropyl)-α-phenylglycine under phase transfer catalysis conditions[200] and studies are reported on the progress in the no-carrier-added radiosynthesis of [^{18}F]-fluoroarginine for use as a probe for nitric oxide synthetase activity.[201]

L-cysteic acid has been synthesised by indirect electrooxidation and its applications have been discussed,[202] and the synthesis and structures of Fe(Cysteine)$_{1.5}H_2O$ and $Na_2[Fe(Cys)_2]H_2O$ have been reported.[203]

4.3 Synthesis of α-Alkyl-α-Amino Acids. – The synthesis of α-methyl-L-tryptophan, from an indolylmethylimidazolidinone using LDA,[204] α,β-dialkyl-α-phenylalanines, *via* direct alkylation of a Ni(II)-complex of a Schiff base of alanine with (*S*)-*o*-[*N*-(*N*-benzylprolyl)-amino]benzophenone with racemic α-alkylbenzyl bromides[205] and (*S*)-cyclohexylglycine, by the hydrogenation of (*S*)-phenylglycine using rhodium on carbon as a catalyst,[206] have been described.

Diethyl α-acetamido α-alkylated malonates[207] and new amino acid, tosyl and phthalyl amino acid derivatives of 3-carbethoxy methyl-7-hydroxy-4-methyl-coumarin[208] have been synthesised. In both cases, the structures of the final products were confirmed.

In the presence of a Lewis acid, a Michael-type reaction of (**18**) with nitro olefins gave good yields of pyrrolo-oxazolones. These compounds were transformed into α-branched serine derivatives.[209]



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4.4 Synthesis of α-Amino Acids Carrying Alkyl Side Chains and Cyclic Analogues. – Considerable interest has been shown in the synthesis of α-amino acids with alkyl side chain and cyclic analogues and a review of the chemistry of one such, 2-aminocyclopentanecarboxylic acid, has been published.[210]

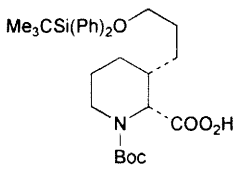
α-Methyl-α-amino acids have been prepared by the Ugi reaction using *Z*-L-Lys(*Z*)-OH, benzylamine, alkyl methyl ketone and cyclohexyl isocyanide, following hydrolysis of the resulting diastereomeric dipeptides,[211] and α-alkyl-α-amino acids were obtained by the hydrolysis of the α-alkyl-α-amino nitriles resulting from the addition of Et_2AlCN and isopropyl alcohol to *N*-sulfinyl imines in an asymmetric Strecker synthesis.[212] α,α-Disubstituted amino acids have been synthesised, also using an asymmetric Strecker synthesis, with alkyl halides or aldehydes,[213] and an efficient enantioselective synthesis of α-methyl-aspartic acid and 3-amino-3-methylpyrrolidin-2-one has been described.[214]

Several groups of workers have conducted studies on pipecolic acid, including the synthesis of C-6 substituted pipecolic acid derivatives using an intramolecu-

lar Mannich-type reaction,[215] the asymmetric synthesis of all four isomers of 4-hydroxypipecolic acid from δ-amino-β-keto esters,[216] the synthesis of a novel constrained pipecolic acid (**19**) in seven steps in 86% yield with 94% optical purity from $TBDPSO(CH_2)_3$,[217] and the preparation of 2,3-methanopipecolic acid from L-lysine *via* 2,3-didehydroo-1,2-bis(methoxycarbonyl)-6-methoxypiperidine. The 6-methoxy group acted as a chiral auxiliary.[218]

The preparation of a series of carbocyclic α-amino acids from four different racemic 2-alkylated cyclopentanones and (*R*)-1-phenylethylamine as the chiral auxilliary by means of an asymmetric Strecker synthesis, the stereoselectivity being influenced by the solvent and by the size of the cyclopentanone C-2 substituent,[219] and the synthesis of novel bridged bicyclic α-amino acid esters (**20**) and key derivatives from quincorine and quincoridine,[220] have been reported.

Stereoselective intramolecular conjugate addition of the benzamide group to cylclohexenone, promoted by Lewis acid and subsequent transformations, has been used to synthesise conformationally constrained hydroxyphenylcyclohexane α-amino acids[221] and 1-aminocyclopropane carboxylic acids and bicyclic α-amino acids have been prepared from a chiral glycine equivalent with a 1,2,3,6-tetrahydropyrazine-2-one structure.[222] The syntheses of α-amino acids with a cyclohexene substituent have been reported[223] and a methodology has been presented for the synthesis and conformational analysis of azacycloalkane amino acids as conformationally constrained probes for mimicry of peptide secondary structures.[224] A protected form of (*R*,*R*,*R*)-2,5-diaminocyclohexanecarboxylic acid has been synthesised and found to function as a building block for helix-forming β-peptides.[225]



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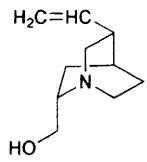

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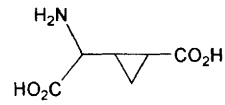

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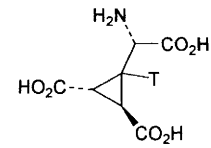

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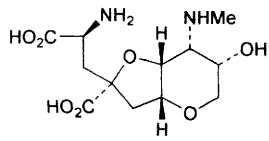

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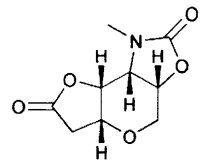

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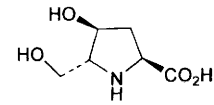

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An improved synthesis of *N*-Boc-*O*-cyclohexyl tyrosine has been reported.[226] The stereoselective syntheses of two carboxycyclopropylglycines (**21**) based on the stereochemical control of the 1,3-dipolar cycloaddition of diazomethane provided by the 4-methyl-2,6,7-trioxabicyclo[2.2.2.]-orthoester function on chiral *E*- or *Z*-3,4-L-didehydroglutamates have been detailed,[227] and the syntheses of (2*S*,1′*R*,2′*R*,3′*R*)-2-(2′,3′-dicarboxycyclopropyl)glycine[228] and (2*S*,2′*R*,3′*R*)-2-(2′,3′-dicarboxycyclopropyl)glycine[229] have been reported. A syn-

thesis of (2*S*,2′*R*,3′*R*)-2-(1′-[^{3}H] ,2′,3′-dicarboxylcyclopropyl)-glycine ([^{3}H]-DCG-IV) (**22**) has also been reported.[230]

Other relevant syntheses reported are those of (−)-dysiherbaine (**23**), a novel neuroexitotoxic amino acid,[231] which has also been synthesised *via* the key intermediate (**24**) which was prepared in seven steps from (2*E*,5*E*)-($PhCH_2CH{:}CH)_2CHOH$,[232] (2*S*,4*R*)-4-hydroxypipecolic acid,[233] the *Z*-isomers of cyclobutane dehydroamino acids from (−)-α-pinene and (−)-verbene,[234] (2*S*,4*S*,5*R*)-(−)-bulgecinine (**25**)[235] and the precursors to vicinal *cis*-dihydroxy-1-aminocyclopentane- and -cyclohexanecarboxylic acid methyl esters which give rise to enantiomerically pure products.[236]

A mixture of the four stereoisomers of *N*-carbamoyl-β-methylphenylalanine was hydrolysed and separated enzymatically to give the four isomers of β-methylphenylalanine in high optical yield[237] and a process is reported for the synthesis of both stereoisomers of 1-amino-4-hydroxycyclohexane-1-carboxylic acid through selective transformations of the functional groups of the corresponding enone cycloadduct provided by the Diels–Alder cycloadditions of Danishefsky's diene to methyl 2-acetamidoacrylate.[238]

Synthetic routes have also been reported for 4-alkyl and 4-cinnamyl glutamic acids, which were subsequently shown to be potent GluR5 kainate receptor agonists,[239] *N*-Fmoc 4-(2′-(di-tert-butyl-malonyl)-phenylalanine – a key step being the introduction of chirality using the Williams auxiliary (benzyl (2*R*,3*S*)-(−)-6-oxo-2,3-diphenyl-4-morpholinecarboxylate),[240] 2-amino-3-hydroxynorbornanecarboxylic acid derivatives containing a conformationally constrained serine skeleton, by cycloaddition of cyclopentadiene with an oxazolylidene derivative,[241] all four stereoisomers of 2,3-methanoleucine,[242] *N*-(2*H*-azirinyl)-L-prolinates which are heterospirocyclic dipeptide synthons,[243] and 2,3-methanoamino acids, prepared from ethyl 3,3-diethoxypropionate by titanium(IV)-mediated cyclopropanation using Grignard reagents.[244]

α-Substituted pyroglutamates have been prepared from tributyltin hydride mediated cyclisation of dehydroalanine,[245] tetralin-based constrained α-amino acid derivatives *via* [4+2]-cycloaddition reaction as a key step[246] and α-CF_3-substituted α-amino phosphonates with two alkene chains, 1,7-dienes and 1,8-dienes have been synthesised by nucleophilic addition to PG-$N{=}C(CF_3)P(O)(OR_2)$. Treatment with a ring closing metathesis catalyst yielded P-containing analogues of dehydropipecolinic and tetrahydroazepin-2-carboxylic acids.[247]

The synthesis of 3,5-di-tert-butyltyrosine from tyrosine ethyl ester by the action of isobutylene in methylene chloride in the presence of sulfuric acid has been described[248] and a general method has been devised for the one-step preparation of 4-(acylamino)piperidine-4-carboxylate esters from the corresponding α-amino acids.[249]

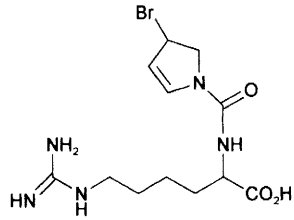
Two procedures for the enantioselective synthesis of protected forms of (3*R*,5*R*)-5-hydroxypiperazic acid have been reported.[250]

The synthesis of α-amino alkanephosphonic acids,[251] and a modified Arndt–Eistert procedure for synthesis of homo-chiral *N*-alkoxycarbonyl α-ethyl aminoadipates and ethyl 6-oxopipecolates have been described.[252] New chiral

amino acids have been synthesised from *cis*-caran-*trans*-4-one and (−)-menthone *via* appropriate lactams.[253]

4.5 Models for Prebiotic Synthesis of Amino Acids. – Theoretical study of the addition of hydrogen cyanide to methanimine in the gas phase and in aqueous solution has been conducted,[254] and the abiotic synthesis of amino acids in simulated primitive environments by radiation has been studied.[255] The stereoselective approach and mechanistic aspects relating to access to proline chimeras have been considered as part of a series of studies looking at pyrrolidines bearing a quaternary α-stereogenic center.[256]

N^{α}-(4-bromopyrrolyl-2-carbonyl)-L-homoarginine (**26**), a natural product from the sponge *Agelas wiedenmayeri*, has been synthesised from lysine. The compound is suggested as a key intermediate in the biosynthesis of pyrrole-imidazole alkaloids.[257]



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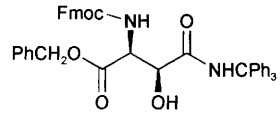
4.6 Synthesis of α-(ω-Halogeno-alkyl) α-Amino Acids. – A new review of the asymmetric synthesis of fluoro amino acids has been published[258] and the review originally published in 1997[259] has been updated.[260] The syntheses of fluoro and difluoroalanines, using tris(diethylamino)-*N*-methylphosphazene for the fluoromethylation of diethyl *N*-acetylaminomalonate by CH_2BrF or $CHClF_2$ in DCM,[261] 3,3-difluoroserine and -cysteine derivatives *via* Mg(0)-promoted selective C–F bond cleavage of trifluoromethyl imines,[262] *cis*-4-[^{18}F]fluoro-L-proline and *trans*-4-[^{18}F]fluoro-L-proline have been synthesised *via* a semi-automated, NCA procedure using the General Electric FDG microlab, a system employing a quaternary 4-aminopyridinium resin to effect F-18 fluorination,[263] and α-difluoromethyl prolines and α-aminoadipic acids by trapping reactions of *in situ* generated *N*-protected α-methyl difluoroalaninyl radicals[264] have been reported.

A facile and stereoselective synthesis of non-racemic trifluoroalanine has been reported.[265] The first synthesis of a totally orthogonal protected α-(trifluoromethyl)- and α-(difluoromethyl)arginine has been reported.[266] The novel synthesis of 5-chloro- and 5-bromo-tryptamines and -tryptophans and its application to the synthesis of bromochelonin has been reported.[267]

4.7 Synthesis of α-(ω-Hydroxyalkyl) α-Amino Acids. – A highly stereoselective synthesis of γ,δ-unsaturated amino acids involving the asymmetrical Claisen rearrangement of allylic esters of TFA-protected amino acids in the presence of

cinchona alkaloids has been reported.[268]

The synthesis of (2*R*,3*S*)-β-hydroxy leucine and all four isomers of β-phenyl serine, using the sulfinimine-mediated Strecker synthesis,[269] 3,4-dihydroxyprolines by application of an L-threonine aldolase-catalysed aldol reaction[270] and (**27**), from methyl (*E*)-4-methoxy cinnamate *via* the Sharpless asymmetric aminohydroxylation reaction,[271] have been reported.



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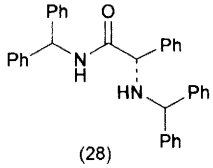
β-Hydroxyaspartic acid derivatives have been synthesised and tested as glutamate transport blockers.[272] On addition of Et_2AlCN/I-PrOH, masked oxo sulfinimines gave α-amino nitriles that afforded oxo α-amino acids on hydrolysis.[273]

4.8 Synthesis of *N*-Substituted α-Amino Acids. – A general route for the solid phase synthesis of *N*-substituted α-amino acids using Fukuyama's sulfonide protecting group has been reported.[274] More specific synthetic methods for *N*-methyl-α-amino acids from *N*-carbamoyl α-amino acids *via* oxazolidinones,[275] *N*-hydroxyamino acids *via* the selective *N*-hydroxylation of *N*-Boc protected primary amino acid esters with methyl(trifluoromethyl)-dioxirane under mild conditions[276] and *N*(σ)-alkyl histamine and histidine derivatives through efficient alkylation followed by deprotection using activated silica gel[277] have been presented. The synthesis of derivatives of arginine containing several chiral centers has been reported.[278]

Reaction of α-amino acids with ketones under hydrogenation conditions using 20% $Pd(OH)_2/C$ gave *N*-monoalkylated amino acids; methylation under the same conditions gave *N*,*N*-dialkylated derivatives.[279] A series of *N*-formyl-*O*-acyl-β-phenylserine derivatives has been prepared by the interaction of *N*-acyl-β-phenyl serine ethyl esters with formic acid in the presence of HF[280] and a series of *O*-(4-amidinophenoxy)alkyl-*N*-substituted tyrosine methyl esters have been synthesised by etherification of 4-cyanophenol with dihaloalkanes in NaOH and conversion of the cyano group to amidine. Their activities were tested against Adp-induced platelet aggregation.[281] γ-Oxygenated *N*-phthalimido glutamic acid derivatives have been prepared by a mild version of the LemieuxJohnson olefin cleavage followed by peroxide mediated dialdehyde oxidation,[282] and the syntheses of (*S*)-proline derivatives which contain a 2,4,6-trimethyl-, 4-tert-butyl- or pentamethylbenzyl-substituent on the nitrogen atom have been reported.[283]

α-Amino amides such as (**28**) have been synthesised by epoxidation of alkylidenedithiane dioxides (R = Ph, 4-ClC_6H_4, 4-$O_2NC_6H_4$, c-C_6H_{11}) *via* the spirocyclic oxiranes (**29**).[284] A number of materials in this group show biological activity. The synthesis has been reported of the complexes of the Schiff bases *N*-vanillin-α-phenylalanine(L_4) with lanthanide(III) ions[285] and these materials have been shown to have antitumour activity. Synthetic routes have also been presented for ^{11}C-labelled *N*-methylaminoisobutyric acid, an achiral synthetic

amino acid which has proved useful for *in vivo* studies of amino acid transport systems in man,[286] N^G-(1-iminoethyl)phosphalysine derivatives, which act as inhibitors of nitric oxide synthase,[287] and *N*-(hydroxyaminocarbonyl)phenylalanine, an inhibitor for carboxypeptidase A.[288]



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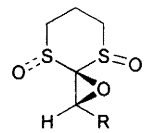

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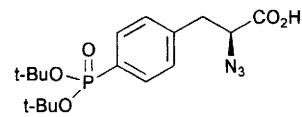

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Also reported are syntheses for N^G-(4-nitrobenzenesulfonyl)-L-arginine,[289] *N*-benzyl-(hydroxyphenyl)glycines *via* a Mannich reaction of phenols with glyoxylic acid and benzylamine,[290] *N*-formamidinylamino acids from amino and formamidinesulfonic acids,[291] *N*-acetyl-L-cysteine, using a new synthetic method using acetic anhydride,[292] and *N*-benzoxycarbony-*S*-phenyl-L-cysteine from L-cysteine, by substituting with benzenediazonium chloride and acylating with CbzCl.[293] 2-Nitrofluoren-9-ylmethyloxycarbonyl amino acids have been prepared by the reaction of 9-fluorenylmethyloxycarbonyl amino acids with 100% nitric acid in DCM,[294] as well as mesityl-substituted amino acids.[295] A large scale production of N^{ε}-trifluoroacetyl-L-lysine, a starting material for the production of lysinopril, is given.[296]

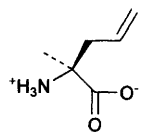
The first synthesis of one of the four possible stereoisomers of 3,4-dihydroxy-L-glutamic acid ((3*S*,4*S*)-DHGA) is reported[297] and four and seven step, respectively, processes are reported for the synthesis of N-Boc-protected (4*R*,3*R*)- and (2*R*,3*S*)-3-fluoroprolines from (2*R*,3*R*)- and (2*S*,3*S*)-3-fluoroprolines.[298]

N^{α}-Lauroylarginyl methyl ester hydrochloride, a cationic surfactant, has been prepared using highly concentrated water-in-oil emulsion as a new reaction media.[299]

A series of novel *N*-[α-(isoflavone-7-*O*-)acetyl]amino acids methyl esters were prepared from chloroacetyl amino acids under mild conditions,[300] *N*-thiazolyl α-amino acids derivatives were readily synthesised from α-amino acids and α-bromo ketones,[301] and the preparation is reported of a new α-azido phosphotyrosyl mimetic (**30**) using a Heck reaction.[302]

The synthesis of the *N*-aryl amino acids has been reported. The coupling of cinnamic and 3-(2-furyl)acrylic acids with amino acids esters followed by saponification and amidation gave rise to *N*-(cinnamoyl)- and 3-(2-furyl)acryloyl amino acids,[303] and the synthesis and biological activity of *N*-aryl-β-alanines and the products of their cyclisation has been reported.[304]

α-Siloxyamides, specifically (−)-betsatin, have been synthesised from H-C(CN)$_2$O-SiMe$_2$.tert-Bu, a carbonyl compound and primary amine, mixed together in acetonitrile,[305] *N*-methylaspartic acid derivatives and their homologues were obtained by a stereoconservative one-pot procedure from hexafluoroacetone-protected aspartic and glutamic acid,[306] and the synthesis of *N*-phosphonamidothionate derivatives of glutamic acid has been detailed.[307]



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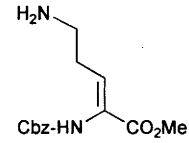

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4.9 Synthesis of α-Amino Acids Carrying Unsaturated Aliphatic Side Chains. – Syntheses have been reported for the α-*C*-methylated side chain unsaturated α-amino acid Mag (**31**), using a chemo-enzymic method,[308] predominantly *Z*-dehydroamino acids from ethyl *N*-Boc- and *N*-*Z*-α-tosylglycinates and nitro compounds,[309] and twenty four 4-alkylidene glutamic acids.[310] The latter were tested as GluR5 agonists. The synthesis of non-proteinogenic amino acids *via* ester enolate Claisen rearrangements is reported.[311]

A synthetic route for the conversion of a (*Z*)-α,β-didehydroornithine (**32**) derivative to α,β-didehydrokyotorphin,[312] the preparation and properties of model dehydroalanine derivatives,[313] and the preparation of α,β-dehydro amino acids, from the reaction of β-hydroxy-α-amino esters with dichloroacetyl chloride in the presence of base,[314] have been reported.

The synthesis of vinyl amino acids is discussed[315] and the installation of the (1-fluoro)vinyl trigger for β,ω-unsaturated amino acids is specifically discussed.[316] A synthetically malleable class of quaternary α-(2-trialkylstannyl)vinyl amino acids that could be used as building blocks in *de novo* peptide design have been described[317] and a generalised synthesis is reported of higher L-α-vinyl amino acids has been given. The side chain is introduced by alkylation of a chiral vinylglycine-derived dianionic dienolate bearing the D'Angelo auxiliary, which can be recovered,[318] and a synthetic route for the preparation of a variety of enantiomerically enriched β,γ-unsaturated α-amino acids by olefination of a Cbz-protected serine aldehyde equivalent has been presented.[319] L-3,4-Didehydrovaline, an important constituent of the antibiotic phomopsin A, has been synthesised from D-serine in 31% yield.[320] The synthesis of γ,δ-didehydrohomoglutamates by the phosphate-catalysed γ-addition reaction to acetylenic esters has been reported[321] and the stereoselective synthesis of *Z*-alkoxycarbonylamino-4-phenylbut-2-enoate is reported.[322] A molybdenum-catalysed regioselective synthesis of α-stannylated allylic esters, suitable substrates for chelate Claisen rearrangements[323] and a one pot reaction of *N*-benzylhomoallylamine with glyoxylic acid monohydrate in methanol to give *N*-benzylallylglycine have been reported.[324] *N*-(5-acetyl-6-methyl-2-oxo-2*H*-pyran-3-yl)-benzamine and *N*-(5-benzoyl-6-methyl-2-oxo-2*H*-pyran-3-yl)benzamide were reacted with various hydrazines to give the corresponding α,β-didehydro-α-amino acid derivatives.[325]

4.10 Synthesis of α-Amino Acids with Aromatic or Heteroaromatic Couplings in the Side Chain. – The synthesis of unnatural amino acids by reduction and ozonolysis of aromatic amino acids has been reviewed,[326] and two reviews on the synthesis of conformationally constrained aromatic amino acids have been published.[327, 328]

A significant body of work has been published regarding the synthesis of α-amino acids with side chains incorporating aromatic groupings in the side chain. The formation of optically active aromatic α-amino acids by catalytic enantioselective addition of imines to aromatic compounds[329] has been reported and an automated synthesis apparatus, developed for L-[3-^{11}C] aromatic amino acids, has been described.[330] The synthesis of indane-based unusual α-amino acid derivatives under phase-transfer catalysis conditions has been reported.[331]

The cross-coupling of aryl boronic acids and alkanethiols mediated by copper(II) acetate and pyridine in anhydrous DMF gave aryl alkyl sulfides; this method can be applied to the synthesis of aryl sulfides of cysteine,[332] while an organoborane, prepared from protected allylglycine, was used in a Suzuki cross-coupling reaction with olefinic aromatic and heteroaromatic bromides to give a range of novel α-amino acids.[333]

The syntheses of optically active phenylglycine derivatives, from *S*-(+)-*N*-(benzylidene)-*p*-toluenesulfinamide using Lewis acids and tert-amines,[334] (*S*)-*N*-tosyl-1-naphthylglycine using a Sharpless asymmetric aminohydroxylation as a key step,[335] and (*S*)-β^2-homoarylglycines[336] have been described. (*R*)- and (*S*)-α-Amino alcohols and α-amino acids, including 4-methoxyhomophenylalanine with a variety of unnatural side chains, were synthesised *via* palladium-catalysed cross-coupling Suzuki reactions.[337] Enantiomerically pure *trans*-cinnamylglycine and -alanine has been prepared by reaction of cinnamyl halides with Ni(II) complexes of chiral Schiff bases of glycine and alanine. The simplicity of the reactions and the high stereochemical outcome make the procedure suitable for large-scale preparations.[338]

The synthesis of 3-(3′-fluorenyl-9′-OXO)-L-alanine, a novel photoreactive conformationally constrained amino acid[339] and photoactivable 4-aroyl-1-phenyl alanines from 4-iodo-1-phenylalanines using a carbonylative Stille cross-coupling reaction[340] has been reported.

Other examples of α-amino acids of this group for which synthetic routes have been reported are all four isomers of α-methyl-β-phenylserine, synthesised from (*S*)- and (*R*)-*N*-Boc-*N*,*O*-isopropylidene-α-methylserinals.[341] The synthesis of α-amino acids with heteroaromatic groupings in the side chain has been reported.

The synthesis of novel heterocyclic substituted α-amino acids using α-amino acid alkynyl ketones as reaction substrates is reported.[342] A number of these have nitrogen-containing rings as part of the side chain. Reactions of 5-substituted (*S*)-1-acyl-3-[(*E*)-(dimethylamino)methylidene]pyrrolidin-2-ones and (*S*)-3-[(*E*)-(dimethylamino)methylidene]tetrahydrofuran-2-ones with amines have been reported. Preparation of intermediates in the ring switching synthesis of hetero-arylalanine- and hetero aryllactic acid derivatives and their analogues have also been detailed.[343] A short and effective enantioselective synthesis of β-heterocyclic amino acid derivatives is described using a kinetic resolution by an acylase from *Aspergillus* species.[344] Syntheses are presented for novel quinolyl glycines, prepared stereoselectively from 2-aminothiophenol and chiral acetylenic ketones which contained a masked α-amino acid functionality, the resulting benzo[*b*][1,4]thiazepine derivatives being converted to quinolyl glycines,[345] phenylalanine and phenylglycine derivatives, possessing a porphyrin moiety,[346]

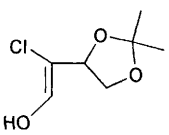
and acetyl-β-(1-azulenyl)-L-alanine in high yield by the malonic ester condensation procedure. This latter compound is a potential blue-coloured fluorescent tryptophan analogue.[347]

Starting from L-serine, pyrazolyloxazolidines have been prepared and transformed into chiral α-amino acids containing a pyrazole ring.[348] The syntheses of β-1*H*-1,2,3-triazol-1-yl and β-2*H*-1,2,3-triazol-2-yl α,β-unsaturated α-amino acid derivatives by an isomerism reaction,[349] a range of novel heterocyclic α-amino acids by the reaction of diamines and amidrazones with α-amino acid vicinal tricarbonyl reactive substrates,[350] and optically active amino acid derivatives of methylated 5-amino-azaheterocycles[351] have been reported.

Cycloaddition of trimethyltin azide with the nitrile group of 4-cyanophenylalanine analogues gave 4-(tetrazol-5-yl)phenylalanine,[352] and pyri-midine and purine amino acids prepared by conjugate radical addition of *N*-(2-iodoethyl)- and *N*-(2-iodopropyl)-pyrimidines and purines with an optically active oxazolidinone[353] have been outlined.

Heterocycles containing both sulfur and nitrogen have also been utilised as parts of side chains. Thiazole- and oxazole-containing amino acids and peptides were prepared using amino acids as educts.[354] The synthesis and resolution of 3-(4-thiazolyl)-D,L-alanine has been reported.[355]

Thiazole containing non-proteinogenic amino acids were synthesised and tested for anti-bacterial activity[356] and orthogonally protected 3-(1-aminoalkyl)isoxazole-4-carboxylic acid has been prepared by 1,3-dipolar cycloaddition of an α-aminonitrile oxide with an enaminoester dipolarophile. The resulting unnatural amino acid, after deprotection, was used as peptide bond replacement.[357] Those analogues containing both oxygen and nitrogen have similarly been used. Analogues of glutamic acid with conformationally restricted structures, 3-carboxyisoxazolinylprolines and related compounds have been synthesised and tested for glutamate receptor activity.[358] A novel isoxazole derivative, *O*-(5-isoxazolyl)-L-serine was synthesised by a Mitsunobu reaction of isoxazolin-5-one with *N*-Boc-L-serine tert-butyl ester and subsequent deprotection of the coupling product is reported.[359]



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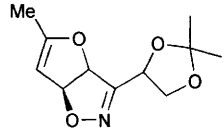

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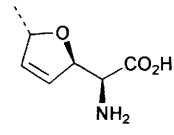

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α-Amino acids with heterocycle side chains containing oxygen have also been synthesised. 1,3-Dipolar cycloadditon of nitrile oxide precursor (**33**) with 2-methylfuran gave the furoisoxazoline intermediate (**34**). This could be converted to four of the eight stereoisomers of L-(+)-furanomycin (**35**).[360] Progress in the synthesis of amino acids containing tetrahydrofuran- and tetrahydropyran amino acids has been reported[361] and 2,2-dimethyl-1,3-dioxane-4,6-dione derivatives of amino acids have been prepared by reaction of the appropriate amino acid with ethoxymethyleneisopropylidene malonate.[362]

Diastereoselective alkylation and/or protonation of chiral enolates have been

used to prepare enanteomerically pure azatyrosine, tribromo- and trichlorophenylalanine[363] and a series of phenylalanine derivatives containing halo-atoms on the benzene ring are reported.[364] Ninety new alkyl/arylsulfonyl and -sulfonylureido glycine hydroxamates have been synthesised and tested as inhibitors of *Clostridium histolyticum* collagenase.[365]

A range of novel heterocyclic substituted α-amino acids has been synthesised by cyclocondensation of (*S*)-2-tert-butoxycarbonylamino-4-oxo-hex-5-ylnoic acid tert-butyl ester with enamines, phenylhydrazine, hydroxylamine and Ph azide.[366]

The syntheses of 3-heteroaromatic-substituted alanines,[367] the optically active phenylglycine derivatives from *S*-(+)-*N*-(benzylidene)-*p*-toluenesulfinamide using Lewis acids and tert-amines,[368] new derivatives of *m*-nitrobenzoyl-D,L-asparagic acid, with potential fungitoxic activity, by the cyclisation of 2-(*m*-nitrophenyl)-4-(β-carboxymethyl)-Δ^2-oxazolin-5-ones have been reported,[369] and some *N*-mustards with esters of *N*-acyl-*m'*-aminobenzoyl-D,L-asparagic acid as supports by ring opening reactions of *N*-acyl-Δ^2-oxazolin-5-ones under the action of di-(β-chloroethyl)-amine[370] are reported.

A convergent synthesis of (*S*)-(−)-3-(2-carboxy-4-pyrrolyl)alanine from a commercially available dimethyl 1-aspartate in good overall yield has been reported,[371] and Fmoc-amino acid azides have been prepared from protected amino acids and NaN_3 by the mixed anhydride method. They are crystalline solids with a long shelf life.[372]

Progress towards the synthesis of fluorodihydroxyphenyl serine has been reviewed and the Evans aldol approach recommended.[373]

4.11 The Synthesis of α-Amino Acids Carrying Amino Groups and Related Nitrogen Functional Groups in Aliphatic Side Chains. – N^{α}-Substituted-N^{β}-protected hydrazinoglycinates have been readily prepared from hydrazines and bromoacetate esters, these materials being useful as potential monomers for solid phase synthesis of hydrazinopeptidoids,[374] and other new potential monomers for solid phase synthesis of hydrazinopeptoids, N^{α}-substituted-N^{β}-protected hydrazinoglycines and hydrazinoglycinals have been identified.[375]

A synthesis is reported of the N^{ω}-hydroxyiminoethyl derivatives of ornithine and lysine. The compounds were tested for inhibition of nitric oxide synthase inactivation[376] and all four *N*,*N'*-protected DAB sterioisomers, using an asymmetric Rh(I)-phosphine-catalysed hydrogenation of isomeric enamides as the key step, have been prepared.[377] An enantiospecific synthesis has been carried out of (*R*)-Boc-(Fmoc)-aminoglycine from (*S*)-Cbz-serine *via* the cyclic carbamate, (*S*)-4-Cbz-amino-2-oxazolidinone.[378]

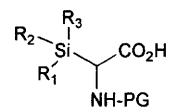
The syntheses of various diamino compounds, namely, (+)- and (−)-2,6-diaminopimelic acids,[379] (*S*,*S*)- and (*R*,*R*)-2-amino-3-methylaminobutanoic acid, from tert-butyl crotonate,[380] and differentially protected (2*S*,4*S*)-2,4-diaminoglutaric acids[381] have been reported. The differentially protected (2*S*,4*S*)-2,4-diaminoglutaric acids were synthesised for incorporation into peptides. Derivatives of N^{γ}-amino-ω-isocyanato-, ω-ureido- and α,ω-diamino acids have been synthesised.[382]

4.12 Synthesis of α-Amino Acids Carrying Boron Functional Groups in Side Chains. – The synthesis of enantiomerically pure ω-borono-α-amino acids of various chain lengths using the general methodology involving the condensation of alkenyl and alkynyl bromides with Ni(II) complex of the Schiff base derived from glycine and (*S*)-2-[*N*-(*N*-benzylprolyl)amino]benzophenone, and hydroboration of the intermediate ω-unsaturated α-amino acids with diisopinocamphylborane, and oxidation with acetaldehyde has been reported.[383] The synthesis of 4-borono-2-fluorophenylalanine, from 4-bromo-2-fluorotoluene[384] and *p*-boronophenylalanine, in six steps from 4-bromobenzaldehyde, has been reported.[385,386] Enantiomerically pure 4-borono-L-phenylalanine has also been synthesised.[387] Studies on the structure of the complex of the latter boron neutron capture drug, with fructose and related carbohydrates, using chemical and ^{13}C NMR methods have also been reported.[388]

4.13 Synthesis of α-Amino Acids Carrying Silicon Functional Groups in Side Chains. – The synthesis is reported of silicon- and germanium- containing α-amino acids and peptides. The synthesised compounds were used to compare C, Si and Ge bioisosterism[389] and β-trimethylsilyl- and -germylalanines have been prepared and studied by single crystal X-ray diffraction.[390]

The synthesis of allylsilane-containing amino acids *via* a Claisen rearrangement has been reported.[391] 3-Trimethylsilylalanine has been prepared enzymatically/microbiologically by two groups of workers.[392,393]

The first synthesis has been reported of α-trialkylsilyl amino acids (**36**, R = Et, CH_2Ph, $R^1 = R^2$ = Me, Et; R^3 = Me, Et, CMe_3, PG = Tos, Boc, Cbz).[394] The synthesis of silaproline, a new proline surrogate, has been reported.[395]



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4.14 Synthesis of α-Amino Acids Carrying Phosphorus Functional Groups in Side Chains. – Readers looking for phosphorus analogues of amino acids should also look in this section.

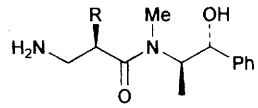
The synthesis is reported of *N*-alkyl-(α-aminoalkyl)phosphine oxides and phosphonic esters, *e.g.* $(MeO)_2P(O)CHPhNHCH(CH_2CHMe_2)CO_2CH_2Ph$ starting from α-amino acids.[396] The synthesis of protected analogues of phosphoserine and their incorporation into peptides has been reported[397] and 1-phosphaserine and 1-phosphaisoserine have been prepared using lipase SP 524. The four stereoisomeric intermediate hydroxyethyl phosphonic acids were separated by capillary electrophoresis with quinine carbamate as the chiral ion pair agent.[398]

A synthesis of the labelled iodinated inhibitor of aminopeptidase N, 2(*S*)-benzyl-3-[hydroxy(1′(*R*)-aminoethyl)phosphinyl]propanoyl-L-3-[^{125}I]-iodotyrosine[399] is reported. The syntheses of *N*-Fmoc-4-[(diethylphosph*ono*)-2,2′difluoro-1′-hydroxyethyl]phenylalanine, as a phosphotyrosyl mimic for the prep-

aration of signal transduction inhibitory peptides,[400] phosphonic analogues of 4-hydroxyproline and 5-hydroxypipecolic acid,[401] and (*S*)-α-cyclopropyl-4-phosphonophenylglycine *via* a multi-step procedure starting from (*R*)-4-benzyloxyphenylglycine[402] have been described.

4.15 Synthesis of α-Amino Acids Carrying Sulfur-, Selenium- or Tellurium-containing Side Chains. – The synthesis has been reported of some Se- and Te-containing amino acids for use as probes for structural studies on proteins.[403] Optically pure amino acids, bearing side chain thioamides, have been synthesised by selective thiations on multiple-carbonyl containing substrates. The products are useful for solid phase peptide synthesis.[404]

Synthetic routes for the preparation of 2-chloroethylnitrososulfamide derivatives of amino acids, from chlorosulfonyl isocyanate *via* carbamoylation–sulfamoylation–cyclisation reactions,[405] and L-selenohomocysteine from L-selenomethionine.[406] The L-selenohomocysteine was used as a substrate for methionine synthase kinetic studies. The cysteine-derived amino alcohol (**37**) has been synthesised as a ligand for iridium(I)-catalysed asymmetric hydrogenation of unsymmetrical ketones.[407]



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4.16 Synthesis of β-Amino Acids and Higher Homologous Amino Acids. – Reviews of diastereoselective approaches to the synthesis of γ-amino-β-hydroxy amino acids[408] and substitution by free radical and anionic chemistry in studies on γ-amino acids and γ-peptides[409] have been published, and a general strategy for the synthesis of the non-natural β^2-amino acids has been described.[410] Synthesis of ω-aminophosphonic acids have been reviewed.[411]

The asymmetric syntheses of β- and α-amino acids have been studied based on carbon radical addition to oxime ethers[412] and asymmetric acyl halide–aldehyde cyclocondensation reactions catalysed by Al(III) triamine complexes gave enantiomerically enriched β-*R*-substituted lactones which underwent ring-opening to give chiral β-amino acids.[413]

The synthesis of substituted β-amino acids has been reported. *N*-substituted β-alanines were prepared by the reaction of 3-amino-9-alkyl carbazoles with acrylic and itaconic acids[414] and *N*-quinolyl-β-alanines have been synthesised by reaction of aminoquinolines and acrylic, methacrylic and crotonic acids, and their biological activity has been investigated.[415] Catalytic enantioselective Mannich-type reactions of silyl enol ethers with aldimes have been performed using a novel chiral zirconium catalyst. The resulting β-amino acids were obtained with high yields and enantioselectivities[416] and the activation of Schiff bases by *N*-glycosylation has been shown to induce asymmetrical Mannich reactions with *O*-silyl ketene acetals to give β-amino acids.[417] β-Amino acids have also been prepared by addition of chiral enolates to nitrones *via* *N*-acyloxyiminium ions.[418]

The synthesis of α-substituted-β-amino acids *via* the amides (**38**, R = Me, Et, Pr, Allyl) has been reported, using pseudoephedrine as a chiral auxiliary,[419] and *via* the aza-aldol reaction of the chiral enolate derived from (2*S*)-*N*-propionylcamphor sultam with *N*-diphenylphosphinyl imines.[420] β-Haloaryl-β-amino acid derivatives have been synthesised using a conjugate addition/oxidative deprotection strategy, employing lithium *N*-benzyl-*N*-α-methyl-4-methoxybenzylamide as a homochiral ammonia equivalent.[421]

α,β-Substitued β-amino acids have been synthesised using a diastereoselective alkylation by organocuprate reagents[422] and by the reaction of *N*-alkoxycarbonyl-1-methoxyamines with optically active 2-oxazolidinones[423] and β-substituted and β,β-disubstituted β-amino acids, which carry a hydroxyalkyl side chain, from sulfonimidoyl functionalised homoallylic alcohols.[424]

Synthetic routes have also been described for the substituted δ-amino acids. α,δ-Disubstituted-δ-amino acids were prepared by stereoselective alkylation of 5-substituted δ-lactams[425] and the first asymmetric synthesis of (*R*)-(−)-α-phenyl δ-amino valeric acid has been reported.[426] The synthesis is reported of 5-amino-4-hydroxy-2,6-dimethylheptanoic acid from *N*-Boc-L-valine methyl ester. The heptanoic acid is a hydroxyethylene isostere of Val-Ala dipeptide.[427]

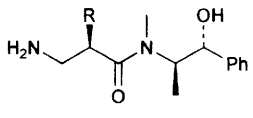
γ-Amino acids and γ-lactams have been prepared from nitro olefins and carboxylic acids using valine-derived 4-isopropyl-5,5-diphenyl-1,3-oxazolidin-2-one as an auxiliary for the enantioselective preparation[428] and *N*-methyl-γ-amino-β-hydroxy acids, essential components of several depsipeptides with interesting therapeutic profiles, have been synthesised *via* a totally stereocontrolled route of preparation.[429]

The synthesis of 'cyclic' amino acids has also been described. These are mainly of two types, the pyrrole-type, where the amino acid nitrogen is included in a ring structure, or the cycloalkane-type, where the amino group and carboxylic acid group are attached to a ring. An asymmetric synthesis of the cyclic β-amino acids generally (**39**, n = 1–3) has been reported.[430]

Syntheses of amino acids of the cycloalkane-type include those of cyclopropane and cyclobutane β-amino acids,[431] diastereo- and enantiomerically pure β-aminocyclopropanecarboxylic acids,[432] 2-aminocyclopentanecarboxylic acid and related alicyclic β-amino acids[433] and methyl (1*S*,2*R*)-1-amino-2,3dihydro-1*H*-indene-2-carboxylate, a new, constrained β-amino ester, using a novel tandem conjugate addition intramolecular electrophilic trap to construct the indane skeleton.[434] Amino acids, incorporating an amino cyclopropyl moiety, have been synthesised by a titanium-mediated transformation of *N*,*N*-dibenzyl-2-benzyloxyacetamide with a variety of alkylmagnesium bromides.[435]

Those of the pyrrole-type include all four stereoisomers of 4-hydroxypipecolic acid, from δ-amino-β-keto esters,[436] (−)-detoxinine, (**40**) the core unit of the detoxifying agent detoxin D_1, from an inexpensive starting material, L-ascorbic acid, *via* the key intermediate (**41**),[437] and a seven step synthesis for the preparation of *N*-benzyl-7-azaspiro[4.5]decane-1-carboxylates (**42**) from 2-oxocyclopentanecarboxylate. The latter are analogues of GABA.[438]

The preparation of an unusual amino acid that mimics a *tripeptide β-strand* and forms β-sheet-like hydrogen-bonded dimers by the condensation of suitably



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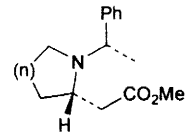

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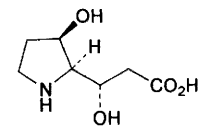

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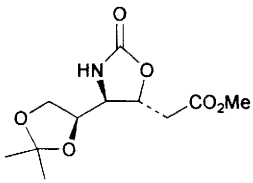

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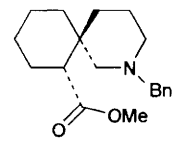

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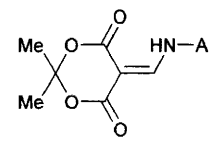

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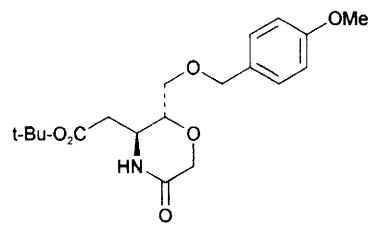

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protected derivatives of hydrazine, 5-amino-2-methoxybenzoic acid and oxalic acid has been detailed.[439]

The syntheses of the methyl esters of the *N*-heteroamino-methylene malonic acids (**43**, A = 2-pyridyl, 2-(5-methylpyridyl), 2-pyrimidyl), which sublime to give oxopyidinopyrimidines,[440] 4-amino-3-(aminomethyl)benzoic acid, in three steps from 4-aminobenzoic acid[441] and *syn*-1-vinyl-2-amino alcohol derivatives by addition of (γ-alkoxyallyl)titaniums with chiral imines[442] have been reported.

Homologation of amino acids has been achieved *via* well-recognised mechanisms. The Arndt–Eistert approach has been used for the synthesis of Boc-/*Z*-/Fmoc-β-amino acids from *N*-protected α-amino acid fluorides in a two-step reaction[443] and *N*-Fmoc-L-β-homoglutamine and *N*-Fmoc-L-β-homoasparagine from *N*-Fmoc-L-α-glutamine and N^{α}-Fmoc-N^{γ}-trityl-L-asparagine.[444] The Michael addition of nucleophiles to *N*-acyl-*N*-(tert-butoxycarbonyl)dehydroalanine methyl ester has been used to synthesise β-alanines[445] and the same technique was used to produce the highly functionalised β-amino acid (3*R*,5*R*,6*R*)-3,6-diamino-5-hydroxyheptanoic acid, the key amino acid of sperabillins B and D,[446] and to synthesise the oxazinone (**44**), which can be alkylated to give protected *anti,anti* α-alkyl β-amino δ-hydroxy esters by Michael addition of the carbamoate moiety of the enoate (*R*,*E*)-$Me_3CO_2CCH{:}CHCH(CH_2OCH_2C_6H_4$-4-OMe)$CH_2O_2CNH_2$.[447]

α-Substituted-β-amino acid derivatives have been synthesised stereoselectively using Wolff rearrangement reactions[448] and the Wolff rearrangement of α-aminodiazoketones derived from N^{α}-urethane-protected α-amino acids that gives rise to the homologation of Fmoc-/Boc-*Z*-α-amino acids to β-amino acids

with concomitant formation of the corresponding pentafluorophenyl esters has been reported.[449]

The route of hydrolysis of heterocyclic rings has also been utilised. 2-Oxazolines have been prepared by $BF_3{\cdot}OEt_2$-catalysed regio- and stereo-selective oxirane ring opening of glycidic esters or amides with MeCN. The oxazolines were hydrolysed into β-amino-α-hydroxy esters or amides[450] and a variety of β-aminoalanine derivatives were prepared by regioselective cleavage of the C(3)–N bond of enantiomerically pure aziridine-2-methanols by nitrogen nucleophiles.[451]

Resin bound *N*-acylated amino acid aldehydes were converted in a single step to α-hydroxy phosphonates by a Pudovik reaction and in six steps to hydroxystatine amides, useful for constructing multiple aspartic acid transition state isosteres.[452]

4.17 Resolution of DL-Amino Acids. – The resolution of DL-amino acids is a key step in amino acid chemistry. This is usually achieved by preferential crystalisation, enzymically, *via* a chromatographic technique utilising a chiral recognition agent, by asymmetric transformation or by absorption onto a polymer/micelle substrate which possesses chiral recognition properties. Papers have been published reviewing the uses of aminoamidases in the enzymic resolution of amino acid amides (84 references)[453] and the separation of enantiomers by gas chromatography (168 references), where amino acids form one of the groups of chiral selector employed.[454]

Preferential crystallisation has been used for the resolution of D,L-α-alanine using L-alanine seed crystal. In this study the addition of OP surfactant was shown to accelerate the crystallisation of L-alanine.[455] The technique has also been used for the optical resolution of D,L-threonine by replacing crystalisation using L-alanine as an optically active co-solute[456] and the erythro- and threo-forms of 4-fluoroglutamic acid through their diastereomeric salts.[457] The phenomena of decrease in purity during the optical resolution of D,L-threonine by preferential crystallisation is discussed.[458]

Crystal structure–solubility relationships in the optical resolution of phenylglycine with (+)-10-camphorsulfonic acid have been studied in detail and the mechanism of the resolving ability discussed.[459]

Enzymic techniques have been used extensively. Enantiomerically enriched β-amino acids have been prepared by enzymic resolution.[460] The enantioselectivity of the lipase-catalysed hydrolysis of amino acid esters has been studied and found to depend on the source of the enzyme, the N-protecting group and the alcohol moiety of the ester[461] and the chiral discrimination of racemic carbazole carbonyl amino acids with linear alkyl side chains by bovine serum albumin was investigated by competitive replacement experiments using dansyl-L-proline and -D-norvaline as fluorescent probes; D-amino acids were bound to the L-proline site more strongly than the L-forms.[462]

Immobilised chymotrypsin on hydrophilic macroporous support has been used for the resolution of D,L-phenylalanine ethyl ester Schiff base. The L-isomer was hydrolysed and the D-isomer recovered unchanged to be hydrolysed chemi-

cally.[463] Horse-heart myoglobin has been shown to promote enantioselective hydrolysis of 4-nitrophenyl esters of amino acids, allowing nearly perfect kinetic resolution of the racemic *N*-Boc-phenylalanine ester (Boc-Phe-ONp)[464] and *N*,*N*-disubstituted α-amino acid phenolic esters have been resolved enzymatically using pig liver esterase on the multi-gram scale and the configuration confirmed by X-ray analysis.[465]

Optically active *N*-benzoyl amino acids have been obtained by the dynamic kinetic resolution of racemic 2-benzyl-4-substituted-5(4*H*)-oxazolones in the presence of an alcohol using *Candida antarctica* lipase B as a catalyst[466] and lipase PS has been used to resolve *cis*-β-hydroxypipecolic acids.[467]

Penicillin G acylase catalysed the acylation of the L-isomers of methyl esters of phenylglycine and derivatives. The process allows the isolation of the enantiomerically pure D-phenylglycine, suitable for conversion into β-lactam antibiotics,[468] and the pure diastereoisomers of 4-fluoroglutamine and 4-fluoroisoglutamine where prepared from the corresponding 4-fluoroglutamic acids. Glutamic decarboxylase treatment of the acids leads to chiral 2-fluoroGABA.[469] A technique for the resolution of *N*-acetyl-D,L methionine methyl ester by protease-catalysed hydrolysis with a mild base as the control agency has been described.[470] Various chromatographic techniques have been used to resolve D,L amino acids and their derivatives. The resolution of basic D,L-amino acids has been effected by direct thin layer chromatography, using a pharmaceutical industrial waste as a chiral impregnating agent,[471] and normal phase TLC has also been used to resolve dansyl-D,L- amino acids on plates impregnated with vancomycin.[472] The resolution of dansyl amino acids, using β-cyclodextrin as a mobile phase additive in reversed-phase TLC, has also been reported, and the effect of structure on the resolution has been studied.[473]

A new π-basic chiral stationary phase has been proposed for the separation of amino acid enantiomers by liquid chromatography. The stationary phase proved especially useful for separating π-acidic *N*-(3,5-dinitrobenzyl)-α-amino amides and esters.[474] HPLC has been utilised to resolve unusual α-amino acids, using direct (Crownpak or Chirobiotic T) stationary phase and indirect methods (precolumn derivatisation)[475] and (1*S*,2*S*)-1,3-diacetoxy-1-(4-nitrophenyl)-2-propylisothiocyanate has been proposed as a new chiral derivatising agent for the HPLC separation of amino acids with two chiral centres.[476] The same group, working with β-substituted tryptophan derivatives, separated all four diastereoisomers by direct (teichoplanin bonded or cyclodextrin bonded stationary phases) or indirect (precolumn derivatisation by chiral reagents) methods. The indirect methods proved more effective.[477] β-Heterocyclic and β-naphthylalanine and phenylalanines were separated by reversed phase HPLC after derivatisation by 1-fluoro-2,4-nitrophenyl-L-valinamide. All L-isomers were eluted faster.[478]

Protonated complexes of amino acids with β-cyclodextrin, produced in the gas phase by electrospray ionisation, were shown to undergo exchange of the amino acid with *N*-propylamine. The rate of exchange varies with the chirality of the amino acid; the enantiomeric excess can thus be determined[479] and copper(II)-assisted enantiomeric analysis of D,L-amino acids using the kinetic method has been studied and the chiral recognition and quantification in the gas phase has

been reported.[480]

Column chromatography (silica gel column) has been used for the resolution of racemic amino acids, using *N*-substituted 2-amino-4-pentenoic acids as a protecting group[481] and the temperature-dependence of the elution order of cyclic α-amino acid enantiomers on copper(II)-*N*,*S*-dioctyl-D-penicillamine ligand exchange column has been studied thermodynamically and a mechanism postulated for the separation.[482] Capillary electrophoresis has been used to separate underivatised amino acids, using copper(II):(*S*)-3-aminopyrrolidine:L-histidine ternary complex as a chiral selector,[483] and the chiral determination of amino acids by capillary electrophoresis and laser-induced fluorescence at picomolar concentrations has been reported.[484]

A study has been reported of the entiomeric separation of *N*-Fmoc amino acids by capiliary electrokinetic chromatography using sulfobutyl ether-β-cyclodextrin as a chiral additive,[485] and chiral analysis has been performed on amino acids in biological solutions by micellar electrokinetic chromatography with laser induced fluorescence detection.[486]

The separation of enantiomers by preparative capillary isotachophoresis, using 2,4-dinitrophenyl-D,L-norleucine as a model analyte has been reported.[487]

Several groups of workers have employed asymmetric transformation as a means of achieving resolution often followed by chromatographic separation. Indirect chiral separation methods based on enantiomeric derivatisations have been developed to monitor optical purity of uncoded amino acids and new series of amino acids using Marfey's reagent for the amino group and (1*R*,2*R*)- or (1*S*,2*S*)-2-amino-1-(nitrophenyl)-1,3-propanediol reagents for the carboxyl group. The diastereomeric derivatives were separated using RP-HPLC and NP-HPLC[488] and homocysteine has been resolved by derivatisation with 4-aminosulfonyl-7-fluoro-2,1,3-benoxadiazole followed by capilliary electrophoresis with γ-cyclodextrin.[489] The validity of the three point interaction model has been examined in the guest exchange reaction involving cyclodextrins and amino acids, and a mechanism for the exchange has been proposed.[490] 4-Hydroxyphenyl- and 4-fluorophenylglycine have been resolved using phenylglycine and (+)-10-camphorsulfonic acid.[491]

A dynamic kinetic resolution of *N*-phthalyl amino acids by stereoselective esterification has been examined using (*S*)-α-methylpantolactone as the chiral auxilliary[492] and the resolution of 1-(2-furyl)-2,2-dimethylpropylamine, an intermediate on a synthetic route to tert-leucine, followed by oxidation, was shown to provide a useful route to (*R*)-and (*S*)-tert-leucine.[493]

The use of polymers and micellular systems to achieve resolution has been reported. A highly enantioselective polymer, imprinted with an organophosphorus compound, was useful for the separation of tryptophan methyl esters[494] and a cross-linked polyvinyl alcohol membrane with L-proline as a chiral ligand has been used for the resolution of amino acids. L-Isomers permeated predominantly through the membrane.[495] The use of ultrafiltration of enantioselective micelles has been shown to provide a low energy, scalable process for the preparation of enantiomerically pure compounds. A model involving the complexation of phenylalanine enantiomers by cholesteryl-L-glutamate anchored in

non-ionic micelles of nonyl-Ph-polyoxyethylene [E10] ether has been reported[496] and a large-scale process for the separation of amino acid enantiomers has been reported in which copper(II)-amino acid derivatives dissolved in non-ionic surfactant micelles were used as the chiral selectors.[497]

A pair of artificial enantiomeric receptors composed of (*S*,*S*)- or (*R*,*R*)-chiral bicyclic guanidinium azacrown ether and (tert-butyl diphenylsilyloxy)methyl group for amino acid zwitterions selectively recognised either L- and D- amino acids[498] and the use of zinc bilinone (the chiral helical dimer of the zinc complex of linear tetrapyrrole) as a chiral recognition agent for α-amino esters is reported.[499]

5 Physico-chemical Studies of Amino Acids

5.1 X-Ray Crystal Analysis of Amino Acids and Their Derivatives. – Crystal structure analysis data have been reported for the following amino acids: D,L-cysteine,[500] D,L-isoleucine and D,L-alloisoleucine,[501] L-arginine phosphate monohydrate,[502, 503] D,L-arginine monohydrate at 100°,[504] L-arginine fluoroborate,[505] ammonium- and methylammonium *N*-acetyl-L-threoninate,[506] *N*-acetyl-L-phenylalanine,[507] sarcosinium trifluoracetate – the N-C-COOH of the protonated molecule is almost completely planar,[508] *N*-methyl-D,L-aspartic acid monohydrate,[509] *N*-methyl-D,L-glutamic acid,[510] L-histidinium dihydrogenarsenate orthoarsenic acid,[511] *N*-benzoylphenylalanine (also solid state ^{13}C NMR),[512] *N*-acetyl-β-trifluromethyl tryptophan ethyl acetate,[513] *N*,*N*-bis(*N*-methylsuccimido) β-alanine, (shows photochromism in its europium-1,10-phenanthroline complex),[514] complexes of maleic acid with L-histidine and L-lysine,[515] the phenylalanine complex of molybdenum-θ-allyl-$(CO)_2$,[516] and other highly derivatised amino acids: *N*-(6-amino-3,4-dihydro-3-methyl-5-nitroso-4-oxopyrimidin-2-yl) derivatives of glycine, valine, serine, threonine and methionine,[517] racemic N^{α}-(-t-butyloxycarbonyl)-L-phenylalanine *N*-methoxy-*N*-methylamide,[518] *N*,*N*-bis(8-hydroxy-5-quinolinemethyl)glycine ethyl ester,[519] 2-methyl-*N*-[(2-nitrophenyl)sulfonyl]alanine and 1-[(2-nitrophenylsulfonyl)-amino]cyclohexanecarboxylic acid.[520] Two conformationally-restricted 4,5-dihydroxynorvaline analogues with a norbornane skeleton,[521] 2,3,5,6,7,8-hexahydro-3-(1-methyl-2-oxopropyl)-6,8-methano-7,7,8a-trimethyl-5*H*-1,4-benzoazin-2-one and its 1-hydroxy derivative,[522] and C-terminal amidated amino acid hydrochlorides.[523]

5.2 Nuclear Magnetic Resonance Spectrometry. – The protonation states of a series of conformationally constrained amino acids (piperidine carboxylic acids) have been studied and correlated with theoretical results from HF/6-31+G* calculations.[524]

The band shape analysis of delayed slow-passage optically detected magnetic resonance has been reported for the photoexcited triplet state of tryptophan.[525] The absolute configuration and enantiomeric analysis of amines and amino acids has been determined using non-chiral derivatising agents and deuterium NMR,[526] conformational equilibrium and intramolecular hydrogen bonding in

nipecotic acid derivatives has been investigated.[527] Solid state NMR has been reported of amino acids and peptides.[528] Caution should be exercised during determination of the absolute configuration of chiral amines by NMR using MPA derivatisation and Ba^{2+} complexation; chemical shifts show inconsistencies with the proposed model relating them to absolute configurations.[529] The characteristics have been determined of the intramolecular H bond in β-alanine, proline, threonine and cysteine by PMR.[530] The rotational isomerism about the C(2)–C(3) bond in aspartic acid and its phosphonic analogues have been studied by PMR. The characteristic vicinal coupling constants were dependent on the populations of the rotamers.[531]

Enantiomeric discrimination in the NMR spectra of underivatised amino acids and α-methyl amino acids has been observed using (+)-(18-crown-6)-2,3,11,12-tetracarboxylic acid as a chiral discriminating agent.[532,533] Samarium(III):(*R*)- or (*S*)-propylenediamine tetraacetate complex has been shown to be useful as a water-soluble chiral shift reagent for use in high-field NMR.[534]

Theoretical studies of the ^{13}C NMR of amino acids has been reviewed.[535]

Both the carboxyl and the hydroxyl ^{17}O resonances of the carboxylic acid group in a tyrosine derivative have been observed for the first time by ^{17}O NMR.[536]

Iodine-127 NQR, IR and X-ray analysis of α- and β-alanine and L-lysine have been reported.[537] NMR studies are reported of the Pt(II) and Pd(II) complexes of glycine[538] and bisalaninates.[539]

Multidimensional variants of the dipolar exchange assisted recoupling (DEAR) NMR have been applied to determinations of ^{13}C-^{14}N dipolar local field spectra in amino acids and peptides.[540] Changes occurring during complexation of praseodymium with serine have been monitored by NMR using spin density matrices.[541]

5.3 Optical Rotatory Dispersion and Circular Dichroism. – The use of optical rotation, CD and other chiroptical properties for the determination of absolute configuration of natural products has been reviewed.[542] The absolute configurations of α-phthalimido carboxylic acid derivatives have been determined from CD spectra.[543] A theoretical treatment has been reported of the photoelectron spectra and CD of L-alanine[544]

5.4 Mass Spectrometry. – Mass spectrometry applied routinely to assist in the structural investigation of amino acids has largely been omitted from this section.

The mobilities of twenty common amino acids have been determined by electrospray ionisation ion mobility spectrometry; not all mixtures of amino acids could be separated by this technique.[545] The fragmentation mechanisms of α-amino acids, protonated under electrospray ionisation, have been the subject of a collisional activation MS and *ab initio* theoretical study.[546] Eight amino acids have been analysed by matrix-assisted laser desorption/ionisation time of flight mass spectrometry and electron-ionisation techniques.[547] Arginine has been shown to form protonated clusters when examined by electrospray ionisation.

This phenomenon has been studied by tandem mass spectrometry.[548] The mass resolved electronic spectrum of cold tryptophan molecules has been obtained by a novel desorption method as a vapourisation source coupled with a supersonic expansion.[549]

The chiral recognition of nineteen common amino acids has been achieved from the collision-induced dissociation spectra of protonated trimers formed from the electrospray ionisation of amino acids in the presence of chiral selectors such as *N*-(tert-butoxycarbonyl)phenylalanine.[550]

Enantiomeric excess of amino acids has been determined by collision-induced dissociation spectra of protonated trimers formed by electrospray ionisation in the presence of a chiral selector.[551] Matrix assisted laser desorption/ionisation mass spectrometry has been used to differentiate isotopically labelled (pseudo-enantiomeric) amino acids using cyclodextrin as a host.[552]

The structures of the fragmentation products of the complex of glycine with zinc(II), produced by electrospray ionisation, have been studied.[553,554]

EI and CI mass spectra of *N*-dinitrophenyl derivatives of amino acids using a particle beam interface show characteristic fragmentation patterns, useful for identifying the amino acids.[555]

5.5 Other Spectroscopic Studies of Amino Acids. – This section covers the other common spectroscopic techniques, which have expanded to cover applications such as conformation determination, in many cases these have been combined with theoretical studies. Theoretical and experimental studies of the vibrational spectra (IR and Raman) of *N*-acetyl-L-alanine[556] and L-valine and L-leucine nitrate[557] have been reported. The IR and molecular structure of zwitterionic L-β-phenylalanine have been determined and compared with the results from *ab initio* calculations.[558]

Conformational studies have included the UV and IR of each of the seven conformational isomers of tryptamine,[559] while gas phase IR and UV ion dip spectroscopy of phenyl alanine has been used to study the most stable conformers.[560] The polymorphic transition of D,L-norleucine from the α-form to the γ-form has been investigated using temperature-scanning time-resolved FTIR.[561] The zwitterions of L-alanine were studied by IR spectra in a KBr matrix, together with the vibrational absorption and vibrational CD spectra. Theoretical calculations were also performed.[562,563] Amino acid salts have also been measured; the IR of sodium and calcium salts of α-amino fatty acids,[564] the FTIR and FT-Raman spectroscopy of D,L-homocysteine and its complexes with Na, K and Ca ions,[565] while in the IR of the monodeuterated salts of tyrosine, valine and some peptides, irradiation in the spectral region produces spectral holes and antiholes resulting from rotation of CD-containing moieties.[566]

Other complexes of amino acids have been studied by IR; the IR and Raman spectra are reported of Cu(II) complexes of aspartic and glutamic acids. The spectra are discussed in relation to their crystal structures.[567] Raman spectra of L-threonine[568] and L-alanine[569] crystals under pressure showed that both underwent a pressure induced phase transition.

The effect of reducing the temperature on the IR spectra of *N*-(tert-butoxycar-

bonyl)amino acids has been reported.[570] The structural changes of amino acids implanted with low energy ions have been studied by FTIR.[571, 572]

Various fluoro-organic compounds, including fluoro-amino acids derivatives were identified in pure and mixed samples by Raman and fluorescence spectra.[573] The colourimetric determination of aromatic amino acids by reaction with 4-chloro-7-nitro-2,1,3-benoxadiazole by measuring absorption maxima at 440–462 nm has been reported.[574]

Analysis has been reported of particle beam-hollow cathode glow discharge atomic emission spectrometry of aromatic amino acids and organomercury and lead compounds,[575] as has the emission spectroscopy of α,ω-diamino acids whose ω-amino group is coupled to a luminescent ruthenium fragment. The α-amino group was protonated. Effect of length of side chain on excited state decay rates has been studied.[576]

X-ray absorption spectra of selenocysteine, selenocystine and sulfo-selenocystine have been compared with the corresponding sulfur K-edge spectra.[577]

Square-wave adsorptive stripping voltammetry has been applied to the study of the interaction of cysteine with monosaccharides at physiological pH. The study was optimised with respect to accumulation time, accumulation potential, scan rate and drop size.[578]

Binding mechanisms and solvent effects have been studied for the molecular recognition of amino acids with zinc porphyrin receptors carrying twelve ester groups.[579]

5.6 Physico-chemical Studies of Amino Acids. – The sub-sections in this chapter have continued with the addition of a new section for measurements of underivatised amino acids in the gas phase.

5.6.1 Measurements for Amino Acid Solutions. Studies of solutions of familiar α-amino acids have lead to the determinations of apparent molar volumes,[580–582] partial molar volumes,[583–586] standard molar enthalpies of solution[587–589] and dilution,[590–594] enthalpies of dissociation,[595, 596] mixing,[597] and protonation.[598] Other properties measured have been viscosity,[599] densities,[600] conductivities,[601–603] solubilities,[604, 605] polarisability, refractive index, solubility and pH and other properties were determined on aqueous L-arginine solutions,[606] dissociation constants,[607, 608] and diffusion coefficients[609]

The effects of amino acids on the crystallisation of other materials hydroxyapatite,[610, 611] calcium phosphate[612] and calcium carbonate[613] have been studied, as well as the crystallisation of some amino acids (metastable crystalline phase of L-glutamic acid (α-form))[614] and single crystals of L-arginine phosphate monohydrate.[615] A study is reported of the crystallisation of glycine and phenylalanine in water-isooctane-AOT microemulsions.[616]

Studies of the solubilities of amino acids with nitrate salts continue; with sodium and potassium,[617] and zinc with histidine, methionine or phenylalanine.[618] Isopiestic studies have been reported on the systems {NaCl + $BaCl_2$ + $mannitol_{(sat)}(aq)$} and {KCl + glycine + $mannitol_{(sat)}(aq)$} at 298.15 K.[619]

Further studies on the gel forming properties of amino acids derivatives have

continued, aqueous gel-like solutions of *N*-acyl-aspartic acids (dodecanoyl–octadecanoyl) formed fibrous supramolecular assemblies which were investigated by atomic force microscopy, small angle neutron scattering and small angle X-ray scattering. The fibres are laterally organised,[620] while aroyl L-cystine derivatives were effective at gelating water.[621] The surfactant properties of different types of derivatives of glutamic acid have been reported.[622]

The effect of cationic surfactants (CTAB and CPB) on the addition–elimination type interaction between aspartic acid and ninhydrin is to increase the peudo first order rate constant.[623]

The characteristics of amino acid extraction from NaCl solutions by reverse micelle using ammonium bis(2-ethylhexyl) phosphate as a surfactant have been reported.[624] A proton transfer reaction, occurring during the extraction of amino acids has been studied using extraction of tryptophan with di(2-ethylhexyl)hydrogen phosphate (D2EHPA) in n-octane and n-octane/n-octanol. In octane, both 1:1 and 1:2 complexes were formed which tended to form clusters. No cluster formation was seen in the more polar solvent system.[625]

Overall partition coefficients of the acid and amine components of amino acid derivatives in an aqueous/organic biphasic system were studied experimentally and theoretically. Partition equilibrium and pH change after partition were predicted by the model.[626]

Studies have been performed on the zwitterions of glycine,[627, 628] *N*-acetylcysteine,[629] aspartic acid[630] and γ-aminobutyric acid.[631]

The kinetics and mechanism of the protonation reactions of amino acids, both inter- and intramolecular, have appeared in several studies; protonation constants,[632, 633] protonation equilibria of L-ornithine and L-glutamic acid in aqueous DMF,[634] the mechanism of interconversion between neutral and zwitterionic forms of glycine has been studied theoretically; proton transfer *via* a water bridge is proposed.[635] The mechanism and energetics of the intramolecular proton transfer of serine in aqueous solution have been reported.[636]

Mechanism of proton transfer from neutral to zwitterionic form of amino acids has been studied.[637] The third order rate constants for the general base-catalysed reaction between *N*-chlorotaurine and its protonated form and for general acid catalysis of the reverse process have been determined. A mechanism for the reaction is thought to involve *N*,*N*-dichlorotaurine as an intermediate.[638]

Proton exchange rates in *N*-acetylglycine have been determined.[639] Formation and stability of the enolates of glycine and its derivatives have been studied. Second order rate constants were measured for carbon deprotonation of the glycine zwitterion, *N*-protonated glycine methyl ester, betaine methyl ester and betaine by D_2O.[640] Solution studies of complexes of amino acids with metal cations have also been reported.

The structure and stability of amino acid phosphonic acid–metal complexes have been reviewed.[641] Complexes can be divided into binary-amino acid only *with metal*; *with copper*,[642–646] with chromium(II),[647, 648] cadmium,[649] vanadium (IV),[650] zinc,[651, 652] and d-block metals.[653]

Ternary complexes contain an amino acid unit and a secondary ligand; the transition metals with an amino acid and γ-picoline,[654, 655] β-picoline and manga-

nese,[656] quadridentate ligands from haloacetylated amino acids and bis(picolyl)-amine then reacted to form trigonal bipyramidal complexes with zinc. The crystal structure of one complex is reported.[657] Complexes have also been studied of amino acids with imidazoles,[658, 659] with sulfamethoxypyridazine,[660] with 2,2-bipyridine[661] and complexation of *N*-(2-nitrophenylsulfonyl)glycine with metals(II) with and without 2,2′-bipyridine in aqueous solution to identify the type, number and stability of the complex species as a function of pH and metal-to-ligand ratio.[662]

Chiral complexes of substituted η^3-butadienyl molybdenum complexes, prepared by reaction of a chlorocarbonyl compound with amino acid esters, were investigated by NMR. Compounds containing one or two stereogenic centres gave rise to dimeric complexes containing dibutadienyl bridging ligands.[663]

Complexes of lanthanum[664, 665] and europium(III)[666] with amino acids and other ligands have also been studied. Paper electrophoresis has been used to study the complexation of dioxouranium with serine and valine. The results suggested complexation through the carboxylate group of the zwitterion.[667]

Other mixed complexes studied have contained proline or 1-hydroxyproline, Cu(II) and an amino acid enantiomer[668] and cystine in the presence of cadmium and folic acid. Adducts are fomed between cystine thiolate and folic acid.[669]

The differential hydration properties of hydrophobic groups of a homologous series of α,ω-amino carboxylic acids were measured in H_2O and D_2O.[670] Time-of-flight neutron diffraction measurements have been carried out on alkaline aqueous 2 mol% glycine solution in heavy water. The hydrogen bonds formed by the amino group nitrogen differ significantly from those formed in neutral solution.[671] A study has been reported of the effects of circulation and facilitated electromigration of amino acids in electrodialysis with ion exchange membranes.[672] An investigation has been reported of the dependence on solvents of optical absorption and emission of a complex of bacteriochlorophyll a with serine.[673]

A voltammetric study is reported of amino acids on gold, platinum, copper and nickel electrodes.[674]

5.6.2 Measurements for Solid Amino Acids. Enthalpies of combustion and formation of eleven aliphatic amino acids[675] and enthalpies of formation crystalline D,L-valine[676] have been reported.

The piezoelectric, dielectric and pyroelectric properties of the twenty protein amino acids have been reported.[677] Surface polarity of α-amino acid crystals has been studied using solvatochromic dyes and compared to poly(amino acids) with the same side chain,[678] and electrostatic properties of α-glycine measured.[679]

Phase transitions have been observed in crystals of D- and L-alanine and valine,[680] and L-alanine.[681] Knoop microhardness anisotropy on the cleavage plane of single crystals of L-arginine hydrochloride monohydrate and the corresponding hydrobromide has been reported.[682] Dislocation resonance damping in L-arginine phosphate monohydrate single crystal has been observed using longitudinal ultrasonic attenuation and velocity measurements.[683, 684] Crystals of (*S*)- and racemic-*N*-benzoylalanine methyl ester had different melting points, reflect-

ing differences in lattice energy.[685] A study has been reported of the refinement and purification of crude glutamine crystal.[686]

5.6.3 Amino Acid Adsorption and Transport Phenomena. Partition of amino acids between immiscible organic and aqueous phases continues to interest researchers; ammonium bis(2-ethylhexyl) phosphate has been used as a reverse micelle surfactant for extracting amino acids from highly concentrated NaCl solutions,[687] the effect of pH on amino acid extraction ratios using AOT reverse micelle,[688] and forward and backward extraction rates of phenylalanine in reversed micellar extraction measured.[689] Extraction and concentration of L-phenylalanine from aqueous solution containing L-phenylalanine has been performed with and without L-aspartate using emulsion liquid membrane.[690] The equilibrium and kinetics have been studied of the extraction of glycine from HCl solutions by reversed micelles[691] and the extraction of amino acids with emulsion liquid membranes using industrial surfactants and lecithin as stabilisers.[692] A new mechanism is proposed for the extraction of amino acids from water to organic solvent using di(2-ethylhexyl)phosphoric acid. The mechanism explains the dependence of the equilibrium constant on the loading ratio.[693] A novel artificial receptor for aromatic amino acid zwitterions, prepared in three steps from a chiral bis(aminomethyl)bicyclic guanidinium salt, allows the aromatic amino acid to move from aqueous solution to DCM.[694] Extraction of amino acids from aqueous solutions into chloroform occurs using di(2-ethylhexyl)phosphoric acid in the presence of dicyclohexyl-18-crown-6.[695] Spectrophotometry and pH measurements have been used to study the extraction of Co(III) and Cu(II) complexes of amino acids from an aqueous donating phase into chloroform liquid membrane containing calix[4]resorcinarene; Cu(II) complexes are extracted more efficiently, especially if the aqueous phase is alkaline.[696] A tryptophan–tyrosine mixture has been separated by non-ion exchange sorption on an anion exchanger with hydrochloric acid. The sorption of the amino acids was temperature dependent.[697]

Studies have been reported of the adsorption of amino acids onto surfaces; onto silk fibroin, and synthetic polypeptides[698] and films; grafting of amino acids onto PET film surface was found to improve the surface properties of the amino acids such as wetability and neg. ion activity for use in medical techniques.[699] Overoxidised polypyrrole films templated with L-glutamate selectively take L-glutamic acid and other L-amino acids into the film.[700]

The thermodynamic functions for the sorption of aromatic amino acids on KU-2x8 sulfocationite in the H-form have been determined.[701] A study is reported of the adsorption and electroadsorption of amino acids from aqueous solution on uncharged and electrochemically polarised carbonaceous material.[702] Adsorption of tyrosine on to activated carbon/water interface has been shown to be pH dependent.[703] Adsorption of glycine and alanine on montmorillonite with or without divalent cations has also been studied.[704]

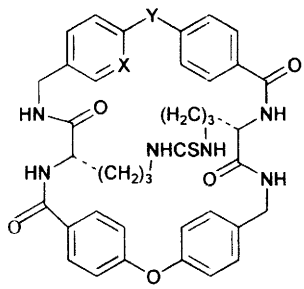
Studies of amino acids adsorbed onto metal surfaces have also been published; the microscopic monolayers of cystine and cysteine assembled on Au(111) form hydrogen bonded cluster networks,[705] adsorption of L-cysteine on gold by elec-

trochemical desorption and copper(II) ion complexation has been studied.[706] A combined density functional theory and X-ray emission spectra study has been reported of the electronic structure and surface chemistry of glycine adsorbed on Cu(110).[707] The adsorption behaviour of aspartic acid on Cu(001), studied by scanning tunnelling microscopy, shows features such as inability to form ordered structures which are different from the adsorption behaviour of other amino acids.[708] The adsorption behaviour of amino acids on a stainless steel surface has been studied.[709]

5.6.4 Host–Guest Studies with Amino Acids. Studies on the complexation of tryptophan and its derivatives with cyclodextrins continue. The 1:1 host guest complexes formed by 6α-(2-amino-ethylamino)-6α-deoxy-β-cyclodextrin and (*R*)- and (*S*)-tryptophan have been studied by pH titrimetric and NMR spectroscopic studies.[710] Organoselenium-containing β-cyclodextrins and their complexes with L- and D-tryptophan were studied by NMR, IR and combustion analyses.[711] Other cyclodextrin studies have appeared; a correlation has been found between the conformation and chiral recognition of a series of amino acid complexes with β- and γ-cyclodextrins using titration microcalorimetry and PMR,[712] and the inclusion complexation behaviour of the methionine, proline and isoleucine derivatives of β-cyclodextrin has been studied by fluorescence spectrometry. The amino acid derivative showed increased binding ability with 8-anilino-1-naphthalenesulfonic acid ammonium salt compared to the parent cyclodextrin, but decreased ability with Rhodamine B.[713] Enantioselectivity towards amino acids by metallo-6^A-deoxy-6^A-hydroxyethylamino-β-cyclodextrin has been investigated by potentiometric titration of the amino acids with NaOH. Nickel complexes show the greatest enantioselectivity.[714] A thermodynamic study is reported of the complexation of γ-cyclodextrin with *N*-carbobenzyloxy aromatic amino acids and ω-phenylalkanoic acids.[715]

The stability constants for the inclusion complexes of *p*-sulfonatocalix[4] arene with amino acids have been measured.[716] The complex of *p*-sulfonatocalix[4]arene with L-lysine shows a cationic substrate spanning the hydrophobic bilayer.[717] Chiral homoazacalixarenes possessing amino acid residues have been prepared. Their preferred conformation was a cone.[718] The rate of alcoholysis of *N*-acetyl-1-amino acids in methanol increased markedly in the presence of *p*-sulfonatocalix[*n*]arenes compared to *p*-hydroxybenzenesulfonic acid; NMR indicated the formation of an inclusion complex between the calixarene and *N*-acetyl-1-histidine.[719] Cryptand[222] undergoes selective complex formation with some polar and aromatic amino acids; thermodynamic functions and equilibrium constants of complex formation were calculated for histidine, threonine and glutamine.[720] Liquid–liquid extraction of non-protein amino acids by 18-crown-6 and cryptand[2.2.2.] shows a relationship with the amino acid structure.[721] Two new receptors (**45**, X = N, Y = O; X = CH, Y = CH_2) have been prepared. The presence of the pyridyl unit provides an additional H-bonding functionality.[722] A new class of C1- or C-2 symmetrical host molecules based on a spirobisindane skeleton has been used for diamines. One host molecule prefers short rigid diamines (lysine), the other longer α,ω-dications

(**46**).[723] Molecular recognition of amino acid esters by 5-(2-carboxyphenyl)-10,15,20-triphenylporphyrinatozinc(II) was investigated by UV–vis spectrophotometric titration method. The host–guest binding mode was studied by PMR.[724] A further study on fixed site heteropolysiloxane membranes containing grafted macrocylic receptors, used to separate mixtures of amino acids, has been reported. A dual transport mechanism is proposed.[725]



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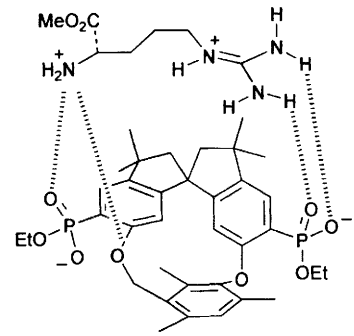

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A liquid chromatography and ultrafiltration study has been reported of the binding of D- and L-tryptophan to bovine serum albumin in the pH range 7 to 11.[726]

5.6.5 Gas Phase Measurements. Studies of cationised glycine and its derivatives in the gas phase have been reported; Gly.M^{2+} (M = Be, Mg, Ca, Sr, Ba) – the divalent metal ions dramatically influence the structure of glycine in the gas phase.[727] The influence of derivatisation, proton affinity and alkali metal addition on the stability of a series of N- and C-methylated glycines cationised by alkali ions[728] and the enol of glycine $H_2N\text{-}CH=C(OH)_2$ generated in the gas phase by neutralisation of the corresponding radical cation has been studied. The reionisation shows that the enol exists and does not isomerise significantly to the more stable glycine.[729]

5.7 Molecular Orbital Calculations for Amino Acids. – For a large range of amino acids, the following properties have been studied; the mechanism of proton exchange between amino acids side chains and water,[730] VAED characterisation (Vector of atomic electronegative distance) and ^{13}C simulation for 20 natural amino acids using MATLAB and True basic programs,[731] solvation free energies (hydrophobicities),[732] selected properties of amino acids have correlated using a variable connectivity index, $^{1}\chi^{f}$, which is obtained by introducing variable weights into a generalised connectivtiy index.[733]

Glycine has featured most heavily in MO calculations with the following; the radiation products of glycine crystals, structures are proposed for the four radicals formed,[734] the potential energy surface of glycine, and the vibrational state and spectroscopy computed from the results,[735] solvent effects on intra-

molecular proton transfer in glycine hydrated by three water molecules,[736] the effect of ionisation on the relative stabilities of the four lowest conformers of glycine and the intramolecular proton transfer process transfer in glycine radical cation,[737] the structure and energetics for the four lowest energy conformers of glycine,[738] the mechanism of fragmentation of protonated glycine in the gas phase,[739] the lattice energies of the three polymorphs of glycine,[740] solvent effects on the energetics and molecular response properties of glycine and alanine[741] mechanism of the mass spectral fragmentation of protonated glycine at low energy,[742] a new solvation model combining discrete and continuous descriptions of the solvent has been applied to the relative stabilities of the neutral and zwitterionic forms of glycine,[743] and the interconversion barriers of glycine and L-alanine conformers.[744]

Other amino acid studies have comprised side chain conformational analysis on two derivatives of asparagine and asparaginamide in their γ_1-backbone conformation,[745] charge density, dipole moment, electrostatic potential and electric field gradients for L-asparagine monohydrate,[746] gaseous neutral and zwitterionic forms of alanine show parity-violation,[747, 748] the correlation time for the reorientation of the methyl side chain in crystalline L-alanine,[749] conformational behaviour of β-alanine zwitterion in aqueous solution,[750] phosphorylation and dephosphorylation of serine, threonine and tyrosine phosphate,[751] proton affinities and gas-phase basicities of glycine, serine and cysteine.[752]

Complexation of cations with amino acids in the gas phase have also been studied; interaction of neutral and zwitterionic glycine in the gas phase with Zn^{2+} ions,[753] zwitterionic glycine bridged with NaCl,[754] cation-σ interactions for complexes of Na^+ and K^+ with aromatic amino acids,[755] gas phase metal ion (Li^+, Na^+, Cu^+) affinities of glycine and alanine,[756] a conformation and hydrogen bonding study of the complex of alaninamide and water. The lowest energy conformer had a network of intermolecular hydrogen bonds from the amide to water and from the water to the carbonyl oxygen.[757]

An *ab initio* analysis has been reported of the stability of different conformers of glycine, *N*-methylglycine and *N*,*N*-dimethylglycine. The effects of solvent and group size on the tautomerisation were studied.[758] The same authors have also reported similar calculations for fluoroglycine and have compared the two sets of results.[759, 760]

Derivatives of amino acids studied have included: the β_{DL} conformer of *N*-formyl-*trans*-2,3-didehydroalaninamide was shown to be the most stable,[761] the geometric and energetic properties of a diamide of serine, HCO-NH-L-$CH(CH_2OH)CO-NH_2$,[762] the conformational preference of acetyl-azaalanine *N*-methylamide,[763] a multivariate calibration method has been reported to determine the chemical composition of binary and ternary mixtures of amino acids based on an Imbrie's Q-mode factor analysis.[764]

6 Chemical Studies of Amino Acids

6.1 Racemisation. – Conditions for enzymic racemisation of D-aspartic acid using on-line coupling of a solid phase extraction column and a ligand-exchange HPLC,[765] and for the production of D-glutamate from L-glutamate using glutamate racemase and L-glutamate oxidase have been described.[766]

The use of racemisation of *N*-(9-(9-phenylfluorenyl))serine-derived cyclic sulfamidates in the synthesis of γ-keto α-amino carboxylates and prolines has also been described.[767]

Mild racemisation conditions using metal complexes have also been reported; the rhodium-catalysed racemisation of *N*-acyl α-amino acids has been reported. The technique will be useful for kinetic resolution processes,[768] and an improved procedure for the racemisation of *N*-acyl α-amino acids uses $Pd(PPh_3)_4$ either as the pre-formed complex or by its formation *in situ*.[769]

6.2 General Reactions of Amino Acids. – *6.2.1 Thermal Stability of Amino Acids.* The stability of selected amino acids under attempted redox constrained hydrothermal conditions has been investigated.[770] The pyrolysis of amino acids has been studied and the recovery of starting materials and the yields of condensation products have been determined; the study aims to shed light on the problem of thermal stability of small biomolecules during their extraterrestrial delivery.[771] The mechanisms of thermal decompositions have attracted interest; the thermo-decomposition of asparamide has been studied.[772] It has been demonstrated that the thermal decomposition of the non-natural amino acid *N*-(tert-butoxycarbonyl)-*p*-fluoro-phenylalanine is slightly different from that of its iodo-analogue in that the dehydration reaction is intramolecular.[773]

6.2.2 Reactions at the Amino Group. Studies on the use of the Fmoc-protecting group continue; a range of N^{α}-protected amino acids have been synthesised using Fmoc as an acylating agent under neutral conditions. The procedure circumvents the oligomerisation that occurs under Schotten-Bauman conditions.[774] 9-Fluorenylmethyl fluoroformate is suggested as a useful reagent for the synthesis of Fmoc amino acids; the products are largely dipeptide free.[775] Fmoc-Serine amide has been prepared by a Schotten-Baumann acylation method from Fmoc-Cl and $H.Ser\text{-}NH_2.HCl$.[776] A simple method for the removal of the Fmoc group has been reported. The method uses catalytic BDU in the presence of aliphatic or polymer-supported thiol.[777]

2-(4-Nitrophenylsulfonyl)ethoxycarbonyl (Nsc) is proposed as a new N-protecting group[778] and the relative merits of the Nsc and Fmoc *N*-protecting groups have been compared.[779] Protection of 3,4-dihydroxyphenylalanine using cyclic ethyl chloroformate is proposed for the hydroxy groups for Fmoc solid phase peptide synthesis.[780] Z- and Boc-Protected amino acids have been prepared using *p*-toluenesulfonyl chloride.[781]

Deprotection of *N*-tert-butoxycarbonyl groups in the presence of tert-butyl esters has been achieved using concentrated H_2SO_4 in t-BuOAc or $MeSO_3H$ in t-BuOAc:CH_2Cl_2. Yields ranged from 70–100%.[782]

The synthesis of orthogonally protected lysine derivatives is reported from lysine and protecting agents DdeOH, ZCl and Alloc-Cl.[783]

Monobenzylation of amino acids occurs at ambient temperature using benzyl chloride in water containing potassium carbonate,[784] while mono-alkylation of *N*-(nitrophenyl sulfonyl) α-amino acid esters under solid-liquid PTC conditions occurred with excellent yields without detectable racemisation.[785] Reaction of L-serine and L-threonine with 2-chloroethanol in aqueous KOH gave *N*,*N*-bis(2-hydroxyethyl)-L-serine and L-threonine.[786]

Maillard reaction compounds have been produced by interaction of amino acids and secondary amines with carbonyls.[787]

Various studies on Schiff base complexes of amino acids have been reported; complexes of cobalt(II), nickel(II), copper(II) and zinc(II) with 2-pyridinecarboxaldehyde and a potentially tridentate amino acid,[788] and some bidentate amino acids are reported,[789] complexes of bidentate Schiff base from *p*-hydroxybenzaldehyde and L-(+)-cysteine;[790] dimethyltin dichloride with amino acid Schiff bases gave 1:2 coordination compounds.[791] The synthesis, mechanism of formation and NMR spectra of lanthanide complexes with an unsymmetrical Schiff base are reported.[792]

Kinetic studies of the interaction of amino acids with aldehydes have also appeared; with vanillin the reactions showed 1st order kinetics.[793] The kinetics of the condensation of glutaraldehyde with amino acids have been studied using UV, pH measurements, microcalorimetry and analysis of functional groups.[794] Reaction of amino acids with *o*-phthalic aldehyde in the presence of sulfite and cyanide ions enabled their determination by spectrophotometric and fluorometric methods.[795]

Studies were reported on the complexes of organo tin(IV) compounds with Schiff bases formed from heterocyclic ketones and amino acids. The resulting compounds were studied by NMR and screened for antibacterial activity.[796]

Various studies of the alkaline permanganate degradation of amino acids have been reported.[797–799]

6.2.3 Reactions at the Carboxy Group. The (2-phenyl-2-trimethylsilyl)ethyl group is proposed as a new carboxy protecting group; the group can be cleaved with tetra-n-butyl ammonium fluoride.[800] The deprotection of t-butyl esters using HNO_3 in CH_2Cl_2 has been investigated. Some unwanted transformations were observed.[801] The electrochemical deprotection of amino acids from their Dim esters is reported.[802]

Boc and Z-Protected amino acid fluorides have been synthesised using DAST as a fluorinating agent.[803]

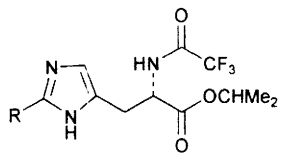
Studies on the decarboxylation of amino acids have been numerous, reported for the reactions themselves, kinetic and mechanistic studies have been reported for the decarboxylation of amino acids by chloramine T,[804] with and without the presence of micelles,[805] and on the role of chlorine in the reaction[806] or for synthetic purposes. A mild and efficient method is reported for the synthesis of 2-substituted pyrrolidinones from amino acids by a tandem radical decarboxylation–oxidation. The reaction proceeds with high yields and good stereoselectiv-

ity.[807] A new synthetic method for the preparation of imides through an oxidative photodecarboxylation reaction of *N*-protected α-amino acids using FSM-16, a mesoporous silica.[808] The anodic oxidation of *N*-acetylisoleucine resulted in a decarboxylation/methoxylation product.[809]

The rate of spontaneous decarboxylation of amino acids has also been studied.[810]

An efficient procedure has been reported for the reduction of α-amino acids to enantiomerically pure α-methyl amines using $LiBH_4$/TMSCl reagent.[811]

A range of imidazoles, including the histidine (**47**), and thiazole (**48**) with chiral side chains derived from amino acids have been prepared from *N*-Cbz-protected α-amino glyoxals. The α-amino glyoxals were obtained from L-amino acids *via* diazo ketones.[812–814]



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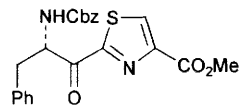

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Kinetic studies on basic procedures have been reported on the esterification of L-phenylalanine by methanol,[815] and on the base-catalysed hydrolysis of amino acid esters in the presence of Cu(II)-complexes with a polymer of glutamic acid and ethane-1,2-diol. The rate is enhanced by the presence of these polymers.[816]

6.2.4 Reactions at Both Amino and Carboxy Groups. Kinetic and mechanistic studies have been reported of the oxidative deamination and decarboxylation of L-valine by alkaline permanganate,[817] of silver(I) ion-catalysed oxidative deamination and decarboxylation of D,L-valine by acidic permanganate,[818] of L-amino acids by potassium permanganate in moderately concentrated sulfuric acid; the latter reaction occurs in a two-stage process, both stages first order[819] and of six amino acids by chloramine T.[820]

A study of isotope fractionation during radiation-induced decarboxylation and deamination of L-leucine showed that was more pronounced for $^{13}C/^{12}C$ than for $^{15}N/^{14}N$.[821]

A facile method for the transformation of *N*-(tert-butoxycarbonyl) α-amino acids to *N*-unprotected α-amino methyl esters is reported.[822]

The Dakin–West reaction of *N*-alkoxycarbonyl-*N*-alkyl-α-amino acids employing trifluoroacetic anhydride is reported.[823]

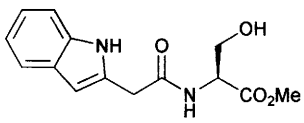
6.2.5 Reactions at the α-Carbon Atom of α- and β-Amino Acids. Other papers under this heading may also appear in the synthesis Sections 4 or in Specific Reactions (6.3), depending on the emphasis of the paper.

The direct asymmetric α-methylation of α-amino acids in two steps has been reported. *N*-protected amino acids were treated with KHMDS followed by MeI in THF/toluene to give high yields with good enantiomeric excess.[824] The stereoselective alkylation of aldimines; prepared from α-amino esters and

pyridoxal models having an ionophoric side chain composed of a chiral glycerol structure; in the presence of Li^+ or Na^+ gave α,α-dialkyl amino esters after acidic hydrolysis.[825]

The treatment of *N*-MOM-*N*-Boc-α-amino acid derivatives with potassium hexamethyldisilazide followed by methyl iodide under low temperature conditions good yields of the corresponding α-methylated products.[826] Reaction of trifluoracetic anhydride with α-hydroxy acids or α-amino acids in the presence of pyridine was a convenient synthesis of α-trifluoromethylated acyloins.[827]

6.3 Specific Reactions of Amino Acids. – For this year's review, in order to obviate the ever swelling size of Section 6.3, which has become something of a 'catch all' section, an attempt has been made to find more specific locations, either in 'Synthesis' or a specific reaction site (*e.g.* Section 6.2) for more of the papers.



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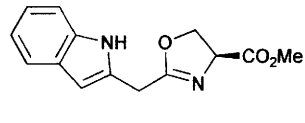

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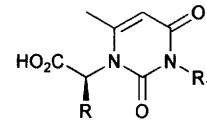

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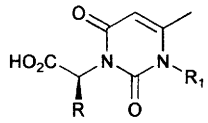

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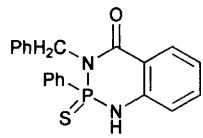

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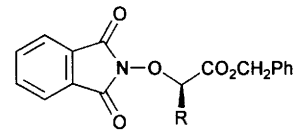

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Reviews of biodegradability characteristics and applications of asparagine acid-[828] glutamic acid-[829] and methylglycine-based chelating agents have appeared.[830] The use of amino acids in the synthesis of heterocyclic compounds continues to prosper. A facile synthesis for heterocycles containing a glycine residue has been reported.[831] A mild and efficient conversion of β-hydroxy amides (**49**) to oxazolines (**50**) is described using DAST and $(MeOCH_2CH_2)NSF_3$ reagents. DAST gives higher yields for serine-containing substrates, whereas $(MeOCH_2CH_2)NSF_3$ gives higher yields for threonine.[832] Z, BOC, FMOC and ALLOC derivatives of 5-aminooxazoles were prepared in one step from acyl amino acids and chlorosulfonyl carbamates.[833] A solid phase procedure, giving a high yield and optical purity, for the synthesis of the uracils (**51**) and (**52**) has been reported, using resin-bound amino acids with isocyanates.[834] A one pot synthesis of the novel 5,11-dioxo-6-methyl-5,9,10,11-tetrahydro-8*H*-naphtho[2,3:1,2]pyrrolizine and its 9-acetoxy analogue[835] and a facile and convenient synthetic method for fluorine-containing 1*H*-pyrrolo[3,2-*h*]quinolines have been reported.[836]

Other novel cyclisation reactions have included an intramolecular defluorinative cyclisation synthesis of difluoromethylated quinazolic acid derivatives,[837] heterocycles of type (**53**) have been produced by the rearrangement, in alcohol, of ester or nitrile derivatives of β-amino acids with the formation of a

β-peptide link.[838] The reaction of aspartic acid derivatives with Grignard reagents yielded γ,γ-disubstituted α- and β-aminobutyrolactones.[839] Optically active β-amino acid *N*-carboxyanhydrides have been synthesised through cyclising N^{β}-Boc β-amino acids using PBr_3.[840] The D-α-(phthaloylamino)oxy acids (**54**, R = iso-Pr, sec-Bu, CH_2Ph, CH_2CONH_2) were synthesised using a Mitsunobo reaction from L-amino acids with inversion of configuration.[841] Papers have reported the conversion of amino acid derivatives to alkaloids or their precursors; *via* *N*-acyliminium ions generated in a one-pot radical decarboxylation–oxidation[842] and heterocyclic-β-amino esters were shown to be diastereoselectively alkylated with alkyl halides to lead to direct precursors of bicyclic alkaloids.[843]

Studies of the reactions of amino acids with other natural products, heterocycles and other compound types have continued. The synthesis of amino acid derivatives of 7-methoxycarbonylneoflavones,[844] optically active derivatives methylated 5-amino-azaheterocycles[845] and naphthalene-1,2-dione-amino acid adducts have been reported.[846] Amino acid-estradiol derivatives have been synthesised enzymatically for the first time using a protease-catalysed condensation.[847]

Complexes of adducts of amino acids with nucleobases and of their model compounds have been discussed.[848] The formation of a *N*-glycosidic linkage between *N*-acetylglucosamine and asparagines, using aspartic acid γ-fluoride in combination with either glycosyl azide or Bu_4NF, has been investigated.[849] Diaryl-selenides and selenones containing amino acid moieties have been synthesised from 4′-nitro-4-aminodiphenylselenide.[850]

The kinetics and mechanism of the reaction between amino acids and stable free radicals derived from 2,2′-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid) have been investigated. The reaction has a three-step mechanism with complex kinetics.[851]

Amino acids are used as ligands or supports in various reactions. The synthesis of polymer supported α-amino acids and their application in the alkylation of arenes has been described.[852] Enantioselective Si–H insertion of methyl phenyldiazoacetate catalysed by dirhodium(II) carboxylates incorporating *N*-phthaloyl-(*S*)-amino acids as chiral bridging ligands has been reported.[853]

Some phosphorus-containing derivatives and analogues of amino acids do not fit snugly into the section on synthesis of compounds with phosphorus in the side chain. They are reported here. 5′-*O*-Derivatisation of AZT with the *O*-methyl esters of phenylalanine and tryptophan gave 5′-amino acid phosphoramidothioates.[854]

Sulfamates, R-X-SO_2-NH_2 (X = O, NH), derived from amino acids, have been shown to react with trialkyl phosphates, in the presence of diisopropylazodicarboxylate, to give phospha-λ^5-azenes which undergo an imidate–amidate rearrangement to yield *N*-phosphorylsulfamates, bioisosteres of pyrophosphate.[855] The synthesis has been reported of *N*-alkyl-(α-aminoalkyl)phosphine oxides and phosphonic esters, *e.g.* $(MeO)_2P(O)CHPhNHCH(CH_2CHMe_2)CO_2CH_2Ph$ starting from α-amino acids.[856] Condensation of allylated amino acids with methyl or vinyl phosphonic dichlorides gave rise to three diastereomeric P-chiral amino

acid-derived phosphonamidic anhydrides. The mechanism of the reaction is discussed.[857] 2-Hydroxy esters of oxophosphorus acid react with glycine to give amidoglycine H-phosphinate and cyclic phosphoamido anhydrides.[858]

The kinetics and products of the thiophosphorylation of histidine have been reported.[859] The Michael addition reactions of *O*-quinone methide, generated thermally and photochemically in water, to amino acids and glutathione to give alkylated products has been reported[860] and that between chiral Ni(II) complex of glycine and 3-(*trans*-enoyl)oxazolidin-2-ones have been reported. The latter show electron donor–acceptor attractive interaction-controlled face diastereoselectivity.[861] Similar addition reactions involving allyl groups, *e.g.* diastereoselective addition of allyl reagents to variously *N*-protected L-alanals,[862] and the reaction of allyl isothiocyanates with amino acids and peptides in model aqueous systems have been studied. The latter reactions are pH-dependent. Mechanisms of the observed reactions are proposed.[863]

Complexes/compounds of amino acids with metals can be divided into two types, those containing an amino acid and the metal and those containing a third component (tertiary complexes).

The coordination chemistry of amino acids with platinum and palladium has been reviewed[864] and the preparation of chiral cyclopalladated liquid crystals from amino acids has been described.[865]

Studies of binary complexes have included the formation of complexes between L-carnosine and Cu(II) and their role as catalysts in the hydrolysis of amino acid esters,[866] synthesis and characterisation of manganese(II), cobalt(II), nickel(II) and palladium(II) complexes of D,L-aspartic acid,[867] a method for attaching organometallics to the C-terminus of amino acids *via* a Pd-catalysed, two step procedure is presented,[868] the preparation and reactions of stannylated amino acids,[869] stereoselective synthesis of ferrocenyl amino acids,[870] synthesis and characterisation of La(III) solid complex with L-hydroxyproline,[871] and gold complexes with glycine, histidine and tryptophan. The antimicrobial activity of the complexes is reported.[872]

The reaction of lysine with 18-molybdophosphate to give a salt formulated as $(Lys)_2H_6[P_2Mo_{18}O_{62}]16H_2O$ has been reported.[873]

The complexation of asparagine by dioxovanadium(V) has been studied and the stability constants measured.[874]

A thermochemical study has been reported of the solid phase coordination reaction of glycine and copper hydroxide.[875] The kinetics and mechanism have been studied of the reactions of bis(guanide)copper(II) with amino acids in aqueous media.[876]

Tertiary complexes of Cu(II) and Zn(II) with 2,2′-bipyridal as a primary ligand and amino acids as secondary ligands are reported.[877] Amino acid-derived organozinc reagents have been coupled with aryl triflates at room temperature using palladium catalysts.[878] The complexation of individual amino acids, and amino acids in general, with various metals has been studied over the time period; specifically, complexation of praseodimium and calcium cations with *N*-benzoyl glutamic acid.[879]

The reaction of glycinatocopper complexes with cinnamaldehydes under

mildly basic conditions gave polysubstituted prolines which can be systematically modified in a number of chemoselective transformations[880] and new chiral ligands derived from (*S*)-leucine for the enantioselective addition of diethyl zinc to aldehydes have been described.[881] The reactivity of peroxo α-amino acid (glycine, alanine, valine and leucine) complexes of molybdenum(VI) towards nitric oxide and carbon dioxide in water solutions has been investigated.[882] The structure of a metallated NCA product and its role in polypeptide synthesis involved in the reactions of α-amino acid-*N*-carboxyanhydrides (NCAs) with organometallic palladium(0) and platinum(0) compounds has been investigated.[883]

The decomposition of an amino acid cupric complex using tetrahydrothiazole-2-thione for the preparation of N^{α}-Boc-N^{ϵ}-Fmoc-L-lysine is reported.[884]

There have been many papers on the subject of oxidation, most of them fairly routine. Summarising, kinetics and mechanism of the oxidation of various amino acids have been reported; L-cysteine, L-cystine and *N*-methyl-L-cysteine by potassium ferrate;[885] D-cycloserine by sodium *B*-bromo-*p*-toluenesulfonamide in acid,[886] L-(+)-aspartic acid by diperiodatonickelate(IV) in aqueous alkaline medium[887] and acidic Mn(III) has been used to oxidise phenylalanine,[888] and L-lysine.[889] A series of papers have appeared on the oxidation of various amino acids by vanadium(V) in a micellar system in the presence of sulfuric acid.[890–894] The kinetics and mechanism of oxidation of α-amino acids by benzyltrimethylammonium chlorobromate,[895] and of methionine by hexamethyltetramine-bromine[896] have been studied. Methionine has also been oxidised by peroxynitrite.[897]

Kinetic studies of the oxidation of cysteine in oxygen-saturated aqueous solution in the presence of Cu(II)-containing polymers,[898] and its autoxidation catalysed by copper complexes have been reported. The latter study indicated that catecholamines stimulated the process.[899]

The electroreflectance (ER) technique has been applied to the study of the electrooxidation of some simple amino acids on a Pt(III) surface in acid medium[900] and an electrocatalytic oxidation reaction at a copper microelectrode has been described, which allows the detection of underivatised peptides and amino acids using sinusoidal voltammetry.[901] The oxidation of protein amino acids by free stable hydrazyl radicals (*e.g.* 2-*p*-phenylsulfonic acid 2-phenyl picrylhydrazyl Na salt) has been studied spectrophotometrically.[902]

The ozone oxidation products of amino acids and small peptides have been identified by Electrospray mass and Tandem mass spectrometry.[903] Diastereoselective sulfoxidation of methionine and cysteine derivatives in supercritical CO_2 shows a dramatic pressure dependence; the major product was found to be 'anti'.[904]

Reduction of amino acids has also been widely reported. A simple method for the reduction of carboxylic acids to aldehydes or alcohols using H_2 and Pd/C[905] and the reduction of amino acids containing a hydroxy side chain to β-amino alcohol and the preparation of their peptide alcohols have been studied.[906] Enantiomerically pure 2-amino alcohols have also been prepared by the reduction of α′-(*N*-Boc)amino β-ketosulfoxides,[907] while *syn*-γ-hydroxy-α-amino acids

have been derived from stereoselective sodium borohydride reduction of γ-oxo-α-amino acids catalysed by manganese(II) chloride.[908] A study has shown that the one-electron reduction of selenomethionine oxide occurs more readily than for its sulfur analogue, methionine oxide.[909]

Asymmetric hydrogenation of unsaturated amino acids has been performed using a new aminophosphine phosphinite ligand derived from ketopinic acid as a catalyst,[910] and rhodium complexes with chiral 4-(diphenylphosphanyl)-1-(dialkylamino)butane in the presence of SDS have been investigated. Stereoselectivity was found to be higher in water than in methanol.[911] The mechanism for the homogeneous hydrogenation of dehydroamino acids has been deduced using information based on kinetic studies and NMR characterisation.[912]

A number of reactions do not fit comfortably into other categories and so they are listed here. An improved method for cysteine alkylation, involving the refluxing the cysteine thiol with the appropriate alkyl bromide in a solution of sodium ethoxide in ethanol, is reported.[913]

Isomeric 4-prolinyl and 4,4-diprolinyl amines have been synthesised from 4-epimeric *N*-Boc-4-hydroxyproline tert-butyl esters.[914] The synthesis of *N*-α-amino aldehydes from their morpholine amide derivatives is reported.[915]

A Mannich-type reaction of imines with *N*-protected amino acid chlorides has been found to give good stereoselectivity (99%) using *N*,*N*-phthaloyl-tert-leucine as a chiral auxiliary.[916]

Various reactions, including β-fragmentation reactions, involving aminyl radicals from amino acids have been reported.[917] The synthesis of aspartic acid derivatives useful for the preparation of misacylated transfer RNAs has been described.[918]

The kinetics have been reported of the reaction of sodium glycinate with benzoyl chloride under inverse phase transfer catalysis.[919]

6.4 Effects of Electromagnetic Radiation on Amino Acids. – Irradiation studies have concentrated this year on alanine; two studies using EPR of irradiated alanine,[920] one concentrating on relaxation rates of stable paramagnetic centres.[921] *Ab initio* and semi-empirical methods were used to model radical formation in L-alanine after irradiation; mechanisms of radical formation were deduced.[922]

An efficient nucleophilic substitution reaction of aryl halides with amino acids under focused microwave radiation has been reported.[923]

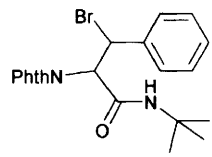
Laser flash photolysis has been use to study a number of reactions; 4-nitroquinoline-1-oxide with D-methionine and its dipeptides have been studied using 248 nm laser flash photolysis,[924] the mechanism of the pyrene sensitised photodecomposition of *N*-phenylglycine depends on the addition of an acceptor as additive.[925] The pH dependence of the photoionisation of aromatic amino acids[926] and the characterisation of transient species of aromatic amino acids using acetone as photosensitiser under laser photolysis have been reported.[927]

A nanosecond laser flash photolysis study is reported of the fast decarboxylation of aliphatic amino acids induced by 4-carboxybenzophenone triplets in aqueous solution. The transfer of protons from aminium radicals within the

solvent cage gives rise to aminyl radicals, which undergo β-decarboxylation. The rate constant for this reaction is an order of magnitude above that observed for the decarboxylation of acyloxy radicals in aqueous media.[928] Supersaturated aqueous solutions of glycine exposed to intense pulses of plane-polarised laser light have been shown to crystallise unexpectedly into the γ-polymorph of glycine.[929]

UV photolysis of protected glycines in the presence of di-tert-butyl peroxide, benzophenone and substituted toluenes lead to selective alkylation at the α-position.[930] The synthesis and characterisation of a photolabile precursor of glycine is reported. The photolysis of the caged-glycine is reported and is proposed as a useful tool for the investigation of the glycine receptor.[931]

The photoionisation characteristics have been reported of amino acids covalently tethered to a naphthol chromophore. The chromophore was separated from the amino acid by an alkyl chain.[932] The photo-induced electron-transfer of ruthenium complexes with derivatised proline residues has been studied.[933] The mechanism of the photolysis reaction of *N*-bromo-*N*-tert-butyl-N^{α}-phthaloylphenylalaninamide to give a 1:1 mixture of the diastereoisomers of 3-bromo-*N*-tert-butyl-N^{α}-phthaloylphenylalaninamide (**55**) is reported.[934]



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The mechanism of pH-dependent photolysis of aliphatic amino acids and enantiomeric enrichment of racemic leucine by circularly polarised light is investigated.[935] An enantioselective fluorescence effect that can be used for determining the optical purity of proline has been reported. The method uses copper(II) complexes of modified cyclodextrins.[936] Fluorescence-quenched ternary complex Cu^{2+}/4-(dimethylamino)benzonitrile/β-cyclodextrin interacted with glutamate to restore the fluorescence.[937] High pressure was found to shift the fluorescence spectra of tryptophan and its derivatives to the red direction, mechanisms for the shift were discussed.[938]

7 Analytical Methods

7.1 Introduction. – Reviews have appeared of the column chromatography,[939] and mass spectrometry and GCMS of phosphorus analogues of amino acids.[940]

7.2 Gas–Liquid Chromatography. – The optimum conditions have been reported for the analysis of amino acid esters by GC using a flame ionisation detector.[941] GC–MS methods for the analysis of stable isotope-labelled amino acids in biological samples continue to attract interests. Methods for the deter-

mination of stable isotope-labelled cysteine and glutathione in biological samples[942] and for the simultaneous determination of isotopic enrichments of ^{13}C labelled homocysteine and methionine in human plasma by GC-negative chemical ionisation MS has been reported.[943] A procedure has been reported for the spectrophotometric determination of aromatic and heterocyclic amino acids in mixtures using the Vierordt method.[944] A GC–MS study has been reported of trimethylsilyl/t-butyldimethylsilyl derivatives of amino acids in model systems.[945]

7.3 Ion-exchange Chromatography. – Amino acid analysis (especially using ion exchange column chromatography) and its relevance to the silk industry has been reviewed.[946]

Amperometric determination has been shown to be useful for the determination of underivatised amino acids at a nickel-modified gold electrode by anion exchange chromatography.[947]

Various theoretical studies of ion exchange chromatography have been reported. Various calculations and theoretical models have been performed on the molecular sorption of amino acids on ion-exchange resins. The calculations were suitable for the prognostication of the selectivity of the ion-exchange sorption of amino acids.[948–950] Electric mass transfer of amino acids through ion exchange membranes has been modelled experimentally by the laser interferometry method. Direct proof has been obtained for the barrier effect in electrodialysis of amino acids.[951]

Ion exchange equilibria of amino acids on strong anionic resins in the hydroxide form have been reported.[952] A study has been reported of the separation of amino acids by displacement chromatography using carbon dioxide as a displacer.[953] A report has appeared of the desalination of a mixture of amino acids using salt-type polystyrene-based strongly acidic cation exchange resin using H_2O as eluant so that the resin did not need regenerating.[954]

7.4 Thin-layer Chromatography. – The thin-layer chromatographic behaviour of twenty-four amino acids was examined on plain silica gel and impregnated with cationic and anionic solutions using water-in-oil microemulsions as mobile phase.[955] A new chiral β-cyclodextrin-bonded stationary phase substituted by 3,5-dinitrobenzoyl groups has been reported for the separation of dansyl amino acid enantiomers.[956]

The TLC properties of sulfur-containing amino acids have been compared with their phosphonic analogues.[957] A reversed phase TLC study has been reported of the interaction of fourteen hydrazines with amino acids and Bovine serum albumin.[958]

7.5 High Performance Liquid Chromatography. – The separation and determination of amino acids in food by HPLC have been reviewed.[959]

The application of LC/MS to the determination of absolute configuration of amino acids has been reviewed.[960]

An HPLC separation system for underivatised amino acids has been coupled

with a fluorescence detection system, giving detection limits of the order of 10 ppm[961] and a system of indirect amperometric detection for these materials for use in microcolumn liquid chromatography has been described.[962]

A procedure has been reported for the determination of amino acids in human blood serum using reversed phase HPLC[963] and HPLC-UV has been used to determine the amount of lysine in a lysine hydrochloride injection.[964]

Chiral HPLC has been used for enantiomeric separation of α-methyl-α-amino acids. Two different methods were employed for derivatised and underivatised compounds.[965] HPLC methodologies using a chiral stationary phase based on the glycopeptide antibiotic teichoplanin has been used for the separation of stereoisomeric cyclic β-substituted α-quaternary α-amino acids,[966] for enantioseparation of *N*-(tert-butyloxycarbonyl)amino acids,[967] for the recognition of amino acids and structurally related compounds,[968] and a procedure for the determination of the chiral purity of synthetic amino acids by HPLC has also been described.[969]

When phenylthiohydantoin derivatives of α-amino acids were separated using polysaccharide-based chiral stationary phases, it was reported that Chiralcel OF preferentially retained D-isomers, whereas Chiralpak AS was better for L-isomers.[970] Enantiomeric and diastereomeric HPLC separation of cyclic β-substituted α-quaternary α-amino acids (cycloalkanecarboxylic acids) was achieved on a copper(II)-D-penicillamine chiral stationary phase. Optimum conditions for the separation of the four possible stereoisomers of each compound in a single run were investigated[971] and the direct HPLC enantioseparation of *N*-protected β-methyl-substituted unusual amino acids on a quinine-derived chiral anion-exchange stationary phase has been described and the effects of different protecting groups were investigated.[972] Various methods of derivatisation have been reported. In order to analyse the various amino acids in rumen fluid, samples were derivatised with 9-fluorenylmethyl chloroformate and separated with a methanol gradient in sodium citrate buffer,[973] *N*-hydroxysucciminidyl-α-naphthyl acetate has been proposed as a precolumn derivatisation reagent for the separation and determination of amino acids by reverse phase HPLC[974] and pre-column derivatisation of amino acids by *o*-phthaldialdehyde/mercaptoethanol and Fmoc with two fluorescence detectors followed by HPLC separation[975]

Methods have been reported for the simultaneous determination of L-phenylalanine and branched chain amino acids in plasma by LC with a co-immobilised enzyme reactor and fluorescence detection[976] and the temperature-responsive chromatographic separation of amino acid phenythiohydantoins using aqueous media as the mobile phase HPLC using modified silica gel with functional polymers. The polymer-grafted surface exhibits temperature regulated hydrophobic/hydrophilic properties changes in water.[977]

The effect of the size of the alkyl substituent on the ester group of benzoyl derivatives of amino acids on the selectivity of the stationary phase (*R*)-3,5-dinitrobenzoylphenyl glycine and binary nonaqueous eluents has been investigated.[978]

Synthetic β-heterocyclic and β-naphthyl alanines and phenylalanines have

been separated on reversed-phase HPLC after derivatisation with FDNP-Val-NH_2. The L-isomers were eluted faster, providing a determination of chiral purity.[979]

7.6 Capillary Zone Electrophoresis (CZE) and Related Analytical Methods. – There is some overlap in this section with the separation of enantiomers of amino acids. Generally, if the emphasis is on the technique, then the paper appears here.

Amendments to the techniques of capiliary electrophoresis have included; sample pre-concentration by filed amplification stacking for microchip-based capillary electrophoresis has shown up to 20-fold signal gains,[980] pressurised gradient capillary electrochromatography for the separation of eighteen amino acids,[981] the development of glass microchips, integrating chemical derivatisations, electrophoretic separations and end column amperometric detections for measurements of amino acids.[982] Amino acids have been separated using planar capillary electrochromatography with an integrated fritless column and conventional stationary phase.[983]

Amino acids have been separated by aqueous two-phase electrophoresis coupled to traditional extraction[984] and using two-phase electrophoresis with dextran-polyethylene glycol-water as a working system.[985] Amino acid enantiomers have also been separated using two-dimensional capilliary electrophoresis coupled to TLC. The TLC plates, which used a mobile phase containing a high concentration of β-cylodextrins, were imaged by laser-induced fluorescence.[986]

A new detection technique exploiting indirect fluorescence has been adapted to the electrophoretic microchip to provide fast analysis of amino acids; sensitivity was lower than previous methods, but the ease of use makes the system attractive.[987] Amino acids and peptides have also been fluorescently labelled, concentrated in organic solvent, separated by CZE and detected by fluorescence,[988] while fluorescein isothiocyanate-labelled amino acids have been separated by capillary electrophoresis with laser-induced fluorescence detection.[989]

N-Dansyl amino acids have been detected on capillary electrophoresis–chemiluminescence analysis using peroxyoxalate reagent. The method had a detection limit of 1×10^{-8} M for *N*-dansyltryptophan.[990]

An on-column derivatisation and analysis of amino acids, peptides and alkylamines by anhydrides has been performed using capillary electrophoresis.[991] An analysis of amino acids from peptide and protein hydrolysates after derivatisation with phenylisothiocyanate is reported using capillary electrophoresis using SDS in phosphate buffer. The results show a 20-fold increase in sensitivity over the HPLC method.[992]

Developments have been reported in the experimental procedures for the chiral separation of amino acid derivatives using capillary electrophoresis. The study indicated that the best experimental conditions varied for each compound analysed.[993] Investigations of the separation of chiral acids[994] and *N*-derivatised amino acids[995] using enantioselective non-aqueous capillary electrochromatography systems have been reported. Chiral separation of amino acids has been achieved by ligand exchange capillary electrochromatography using continuous

beds.[996] Various chiral selectors have been applied to the separation of enantiomers by capillary electrophoresis. Amphiphilic aminosaccharide derivatives have been used as chiral selectors in capillary electrophoresis; their selectivity differed for dansyl amino acids[997] and teichoplanin has been applied to a background electrolyte for enantioseparation of *N*-(tert-butyloxycarbonyl)amino acids as well as to the stationary phase on HPLC (see also Section 7.5).[998] Enantioseparation of anionic analyates by non-aqueous capillary electrophoresis using quinine and quinine derivatives as chiral counter ions for *N*-protected amino acids using benzoyl, nitrobenzoyl and nitrobenzyloxycarbonyl protecting groups has been reported.[999]

Overlapping peaks of amino acid derivatives in capillary electrophoresis have been resolved using multivariant curve resolution based on alternating least squares.[1000] Phenylalanine, isoleucine and tyrosine derivatives of 1,2-naphthoquinone-4-sulfonate were found to be only partially separated by capillary electrophoresis. Partial least squares regression overcomes lack of selectivity for these amino acids. Histidine and leucine derivatives were not separated.[1001]

7.7 Assays for Specific Amino Acids. – The methods of determination of total homocysteine in plasma have been reviewed.[1002] Automatic immunoassay for total plasma homocysteine using a competitive fluorescent polarisation technique has been reported.[1003] L-Cysteic acid has been analysed by reversed-phase HPLC.[1004] An assay is reported for 3-nitrotyrosine in biological tissues and fluids using combined liquid chromatography and tandem mass spectrometry. It is reported that under appropriate conditions 3-nitrotyrosine is formed as an artefact in sample extraction and derivatisation.[1005]

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